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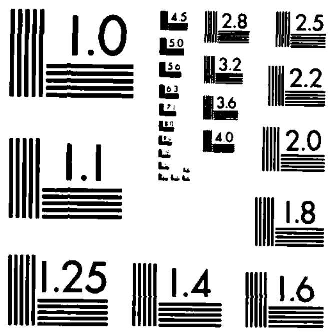

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The second and final phase of the Project Report covers the activities during the period of April 1, 1984 through February 14, 1985.

The first phase of the experimental investigations presented in the earlier Progress Report established that the properties of SET-45 formulas and their modifications appeared to be the most promising for achieving the given objectives of this project. Therefore, the purpose of the second and final phase of the investigation reported here was to test SET-45 formulas and their modifications under all combinations of temperatures simulating summer and winter weather conditions and to establish which one of these materials are the most suitable for emergency repair of concrete runways under various weather conditions.

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The following five materials were investigated:

SET-45 cold weather formula (SC)
SET-45 hot weather formula (SH)
1:1 blend of SET-45 cold and hot formulas (SCH)
SET-45 cold weather formula + 0.34% borax (SCBA)
SET-45 cold weather formula + 0.70% borax (SCBB).

A combination of mechanical and physicochemical examinations were used. The tests have revealed that SET-45 hot weather mortar of flowing consistency appears to be the most suitable for the emergency repair of runways under all weather conditions because it combines the longest observed setting time with the highest early and late age compressive strengths (more than 2000 psi compressive strength at the age of 1 hour) along with satisfactory flexural and bond strengths and volume stability.

The physicochemical examinations revealed that the crystalline part of the hydration product is made up of ammonium magnesium orthophosphate hexahydrate (hexa) and of similar monohydrate (mono), respectively. Mono is the main crystalline product when the hydration is rapid, hexa is the main product when the hydration is slow. The mono-hexa ratio may also increase in SET-45 cold weather mortars later with high curing temperature and dry environment, and decrease with low curing temperature and wet environment due to recrystallization. The quality of the hexa crystals is also affected by temperature because they may grow irregularly, resulting in strength reductions, when the hydration rate is high.

Simple and rapid construction technique is needed when working with SET-45 cements due to the shortness of setting times. Steps of such a method using flowing mortar is described in the report.

The present laboratory results should be supplemented by appropriate field tests.

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PREFACE

This document is a Final Report on the project entitled "Materials for Emergency Repair of Runways". The project identified as No. 83-NA-144 by the Air Force Office of Scientific Research (AFSC) is sponsored by the same Office. It started on October 1, 1983 and ended on February 14, 1985.

The project made use of staff and facilities of the College of Engineering, Drexel University, Philadelphia, PA, 19104. The principal investigator is Dr. Sandor Popovics. Dr. M. Penko has performed the physico-chemical tests as Research Associate and Mr. N. Rajendran has performed the mechanical tests as Research Specialist.

EXECUTIVE SUMMARY - SECOND (FINAL) REPORT

MATERIALS FOR EMERGENCY REPAIR OF RUNWAYS

Prepared for the Air Force Office of Scientific Research

by

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Philadelphia, PA 19104

Scope

The primary objective of this project was to identify or develop an inorganic cementing material that is suitable for emergency repair of damaged airport runways under war conditions.

In the first half of the work several commercially available rapid-hardening cements were screen-tested as presented in ~~our~~ an earlier Progress Report. It was established on this basis that the SET-45 formulas and their modifications appeared to be the most promising for achieving the given objectives of this project. These objectives were:

at least 2000 psi compressive strength at the age of 1 hour;
adequately long setting time;
good bond to old concrete; and
minimum shrinkage under every weather condition.

Since then both mechanical (compressive and flexural strengths, bond, shrinkage, etc.) and physicochemical tests (X-ray diffraction, scanning electron microscopy, infrared spectroscopy, etc.) were performed with these materials to see the technically important properties of these cements under various curing conditions and learn

about the basic nature of these materials. Keywords: hydration; magnesium cement; setting;

The project also has important applications in civilian structures, such as repair of highways, bridgedecks and marine structures. Nevertheless the investigation presented here has concentrated only on the fulfillment of the pertinent needs of the Air Force and omitted research on the civilian aspects of the project.

This Report is the continuation of our Progress Report dated April 30, 1984; that is, it covers the activities during the second and final half of the project during the period of April 1, 1984 through February 14, 1985. The two Reports together form a unit containing all the pertinent tests, results, conclusions and recommendations.

Method of Procedure

The intention of the investigation reported here was

(a) to test two rapid hardening materials, namely SET-45 cold weather and SET-45 hot weather formulations (see: Description of the Materials), and some of their combinations as well as modifications under low and high curing temperatures simulating winter and summer weather conditions;

(b) to establish which ones of these materials are the most suitable for the fulfillment of the requirements for emergency repair of concrete runways under various weather conditions.

More specifically, the following rapid hardening materials were investigated:

SET-45 cold weather formula, marked as SC

SET-45 hot weather formula, marked as SH

1:1 blend of SET-45 cold and hot weather formulas, marked as SCH

SET-45 cold weather formula modified by the addition of 0.34% borax, marked as SCBA

SET-45 cold weather formula modified by the additon of 0.70% borax, marked as SCBB.

All the materials were tested

(a) at high curing temperature with or without precooling the component materials,

(b) at low curing temperature with or without preheating or precooling the components, and

(c) at normal curing temperature but in humid environment.

Test results related to dry curing at normal (approximately 73°F) temperature were reported in our Progress Report earlier.

Description of the Materials

According to the manufacturer (Masterbuilders), the SET-45 mixtures come in two formulas to cover all weather conditions. One is the "cold" formula which is recommended by the manufacturer for cold and regular weather conditions. The other is the "hot" formula for hot weather conditions. Both formulas are granular materials consisting of a powdery cementitious material and sand in the proportion of 1:4 by weight. The cementitious material is a blend of magnesium oxide (MgO) and ammonium dihydrogen phosphate ($NH_4H_2PO_4$) with a small amount of flyash. This blend reacts in the presence of water rapidly producing strength and heat. The hot weather formula also contains boric acid as set retarder.

Mechanical Testing

A major portion of the mechanical testing was devoted to the strengths of various SET-45 mortars at various ages under eight different sets of temperature conditions. Details of these temperature series are presented in Table 1. Each set, or series, contains several, usually five, SET-45 mixtures marked SC, SH, etc., as described under Method of Procedure.

Examples for the setting times and strength development of the five SET-45 mortars at low and high temperatures, respectively, are given in Tables 4 and 5. Compressive strengths after curing at normal temperature are shown in Figure 3. The results of the mechanical tests of the First Series are summarized in Table 15. Flexural strengths and bond strengths are presented in Figure 14 and Table 12, respectively. It can be seen that most of the tested SET-45 mortars had very good strength development not only at temperature around 70°F but also at temperatures representing summer and winter climates, respectively.

A comparison of the experimental results obtained on the newly tested materials indicates that SET-45 hot mixture seems the best for the emergency repair of concrete runways (Table 17) because: SET-45 hot weather mortar requires the least amount of mixing water to achieve flowing consistency; it produces the longest setting times even at elevated temperatures; it develops less heat during setting and hardening than the others; it has the highest compressive strengths; it develops satisfactory flexural strength and bond to hardened portland cement conrete; and it has almost no shrinkage or expansion at early

ages.

It is also clear that special, rapid construction technique should be used for the repair work due to the relatively short setting times of SET-45 mortars. The use of mortar of flowing consistency is a step in this direction because it eliminates the need for compaction.

Physicochemical Investigations

Although the mechanical and physicochemical tests were performed concurrently, most of the time not the same specimens were used for the two tests. The nature of the physicochemical tests requires that, with a few exceptions, only the portion of a SET-45 mixture be used that passed sieve No. 200. This sieve retains most of the sand and flyash which are part of SET-45 mixtures. Therefore the passing material is referred to as SET-45 cement and SET-45 paste, respectively.

The hydration processes of SET-45 cements at normal curing temperature were described in our earlier Progress Report. In the present report mostly the effects of higher or lower than normal temperatures are discussed on the hydration process. Essentially the same five mixtures were used in the physicochemical examinations that were subjected to mechanical testing mentioned above. The water-cement ratio was 0.525 by weight in every case which corresponds to 10.5% water content in the mortar.

A general feature of all five SET-45 combinations cured at elevated temperatures (40°C) is that the main hydration products are similar to those observed at curing at room temperature (Figs. 33 through 52) as presented in the Progress Report. These are:

ammonium magnesium phosphate hexahydrate ($NH_4MgPO_4 \cdot 6H_2O$) and ammonium magnesium phosphate monohydrate ($NH_4MgPO_4 \cdot H_2O$). These are referred to as hexahydrate and monohydrate, or hexa and mono, respectively. In all the mixtures the monohydrate was present in larger quantities at this curing temperature.

In addition to the similarities, there are also certain differences between the hydration processes at normal and at high curing temperatures. For instance, the mono/hexa ratio slowly decreases with curing time at normal temperature but it increases at elevated temperature. Another difference revealed by the comparison of Figures 41 through 48 to Figures 33 through 40 and 49 through 52, is that a characteristic diffraction peak of the hexahydrate at $2\theta = 32.5^0$ ($d = 2.67Å$) is either totally missing or reduced in the X-ray patterns of the three fastest hydrating cements out of the tested five SET-45 cements when cured at elevated temperature. The explanation of this phenomenon is irregularity in the crystal growth at high rate of hydration. This explanation is also supported by the observation that SET-45 hot weather mortar with its slower rate of hydration develops higher strengths than the comparable but faster SET-45 cold weather mortar.

Infrared (IR) spectra of all these cements are identical with the one in (Figs. 53 through 57) indicating no changes in the molecular structures of the hydration products at elevated curing temperature.

The same five mixtures were tested at <u>low curing temperature</u> (0°C) as at high temperature. The general characteristic of these

hydrated pastes is that the X-ray diffraction patterns of all five mixtures are practically identical at various ages after low temperature curing showing only hexahydrate as the crystalline product. The virtual equality of these X-ray patterns is explained by the very slow hydration rate which prevents the development of major differences. It was also shown that SET-45 mortars precooled to 0°C before mixing but cured at 40°C after testing still had low rate of hydration. (Figs. 128 through 138) This seems to indicate that the temperature of the mortar during casting is more important for the hydration process than the temperature of the subsequent curing.

Harmful effects of wet curing of SET-45 mortars has been revealed again by strength reductions. (Table 16) The new physicochemical experiments also indicate such harmful effects. It was found, for instance, that the MgO phase hydrates completely in we.-cured specimens forming brucite, that is, $Mg(OH)_2$, in contradistinction to dry-cured specimens, where a large part of MgO remains unhydrated even after one month. This is shown not only by the comparable X-ray diffraction patterns (Figs. 77 and 78) but also by SEM pictures. (Figs. 73 through 76) The compositional differences, especially in the amorphorus phase of the hydration products, appear also in IR spectra. (Figs. 83 through 86) It is quite likely that the excess amount of $Mg(OH)_2$ creates a physical or chemical instability in the hardened SET-45 pastes causing, among others, strength reductions.

The hydration characteristics of the different SET-45 pastes cured at various temperatures are summarized in Tables 18 through 22.

Conclusions and Recommendations

1. Test results of the mechanical and physicochemical examinations have led to the conclusion that SET-45 hot weather mortar of flowing consistency appears to be the most suitable inorganic cementing material for emergency repair of concrete runways under all weather conditions. The reason for this is that it combines the longest observed setting time with the highest early compressive strengths (over 2000 psi at the age of 1 hour) along with satisfactory other properties, such as good flexural and bond strengths.

2. The hydration of the $NH_4H_2PO_4$ portion of SET-45 cements is always complete in contrast to the hydration of the MgO phase which is almost always incomplete. The crystalline part of the hydration product is made up of ammonium magnesium orthophosphate hexahydrate and the similar monohydrate, respectively. Monohydrate is the main crystalline product when the hydration is rapid, hexahydrate is the main product when the hydration is slow. The mono-hexa ratio may also increase in SET-45 cold mortars later with high curing temperature and dry environment, and decrease with low curing temperature and wet environment. The quality of the hexa crystals is also affected by temperature because they may grow irregularly, resulting in strength reductions, when the hydration rate is high. The temperature of the mortar at casting seems to have greater influence on the rate of hydration of SET-45 cements than the

temperature of the subsequent curing.

3. The construction method used with SET-45 mortars should be quick and simple. For instance, when the runway damage is in the form of a large crater, the major steps of a construction technique may be, as follows:

(a) Push the broken pieces of the damaged pavement as well as those of the base course and subgrade back to the crater;

(b) if this does not fill up the crater completely, add enough crushed stone to it;

(c) compact this loose mass, for instance by a roller;

(d) pour liquid mortar of SET-45 hot weather mixture on the top until the voids in the compacted but still granular mass are filled up;

(e) finish the surface.

The mortar temperature should be close to 70°F during construction regardless of the weather conditions. Plenty of water should be on hand for cleaning equipment. It is also advisable to keep borax powder on hand. In case that the mortar cannot be poured out of the mixer before setting, the borax should be poured into the mixer and mix it with the mortar for the prevention of ruining the mixer.

4. The presented laboratory results should be supplemented by appropriate field tests.

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1. INTRODUCTION

1.1 General

The primary objective of this project was to find an inorganic cementing material that is suitable for emergency repair of demaged airport runways under war conditions. Although practicality required the use of commercially available cementing materials, considerable new research was needed for this project for two reasons. First, not only are the rapid hardening materials new with little practical or scientific experience to rely on but also they are to be used for a special, new purpose, namely emergency repair possibly with a novel construction technique. Also, attempts were made to improve, if necessary, the properties of the commercially available materials with modifications by chemical admixtures.

Therefore, the investigation has been performed in two directions: (a) production of information about the basic nature of rapid hardening cements with or without chemical modifications by using scientific methods, such as X-ray diffraction, scanning electron microscopy, infrared spectroscopy, etc.; and (b) performance of laboratory mechanical experiments to determine the technically important properties of these cements.

On the basis of the reviewed literature four promising rapid hardening materials were selected for screening tests. These materials were SET-45 cold formula, SET-45 hot formula, aluminum phosphate (AℓP) cement, and jet cement. An array of scientific and engineering screening tests was performed on these products to

establish which cement is the most promising for the specified emergency repair. The first phase of the experimental investigations presented in details in our Progress Report established that the mechanical properties of SET-45 formulas by far excelled the others. Therefore, the second phase of the investigation presented here concentrated on the in-depth investigation of SET-45 formulas. More specifically, this work focused on the chemistry of hydration and mechanical performance of SET-45 mixtures at normal, high, and low temperatures that commonly occur during different seasons of a year. In addition, several additives were investigated such as iron salts, silanes, and polyphosphates to improve the mechanical properties of SET-45 mixtures.

The project is obviously U. S. Air Force oriented, nevertheless it has important applications in civilian structures. Examples for this are the use of the obtained results, or their extensions, for repair of potholes in highway pavements, or for overlay on deteriorating bridge decks. These aspects, however, were not investigated in this project.

This Final Report covers the activities during the second half of the project during the period of April 1, 1984 through February 14, 1985.

1.2 Objectives

The specific objectives of the research were the same as stated in the Progress Report; that is, to find an inorganic cement with the following properties:

a. Development of at least 2000 psi compressive strength in one hour, even in cold weather.

b. Development of a sufficient flexural strength.

c. At least 10 minutes of time of initial set, even in hot weather.

d. Sufficient workability of the fresh mixture, preferably flowing consistency.

e. Sufficient bonding capability of the new mixtures to old concrete.

f. Negligible shrinkage to avoid bond failure and cracking.

g. Sufficiently high and permanent final strength.

h. Reasonable cost of the finished repair.

As reported, SET-45 mixtures met almost all the requirements at normal temperatures even with flowing consistency. Therefore, the second phase of the project was to establish if the mix designs judged satisfactory at normal temperature meet the requirements under different weather conditions, that is at elevated temperatures (40°C), at low temperature (0°C) and in humid environment.

Another objective was to obtain a better insight into the hydration of SET-45 mixes under extreme weather conditions by physicochemical tests. A final objective was to improve the setting time as well as strength of SET-45 mixture by using chemical admixtures.

1.3 Scheduling

The mechanical and physico-chemical tests were run concurrently most of the time. Tests at elevated temperatures were performed during the spring and summer and tests at low temperatures during

fall and winter.

2. DESCRIPTION OF THE MATERIALS

2.1 SET-45 Mixtures

New shipments of the two SET-45 mixtures were used in the experiments reported here. The compositions were not rechecked because the manufacturer said that they were the same as those of the SET-45 mixture used in the first half of the project.

SET-45 mixtures come in two formulas in order to cover all weather conditions. One is the "cold" formula and recommended for regular and low temperatures; the other is the "hot" formula and is for hot weather conditions. Both are water activated (hydraulic). These products are commerically available granular materials delivered in 50 lb paper bags. They are manufactured by Master Builders in the plant of SET Products, Inc., Macedonia, Ohio. The product was designed for fast repair of patching potholes. The manufacturers claim that both formulas when properly used, produce strength that exceeds 2000 psi in an hour yet allow enough time for placement.

Both mixtures are light gray, granular materials. The manufacturer does not specify the compositions of the products. Our own findings, discussed in detail in the Progress Report are that the product is a mixture of a powdery cementitious material and sand in the proportion of 1:4 by weight.

The cementitious material is a powder consisting of magnesium oxide (MgO) and ammonium dihydrogen phosphate ($NH_4H_2PO_4$) with a small amount of fly ash. These react with water rapidly producing setting, strength, and heat. Also, the hot weather formula contains boric acid as a retarder.

This report will refer to these two formulas in dry, granular state as "SET-45 cold mixture" and "SET-45 hot mixture". When water is added to a mixture it is called "mortar" instead of mixture. A third term used in this report is "cement". This is the portion of the mixture that passes No. 200 sieve. The mixture of cement and water is called "paste."

2.2 Chemicals

Borax - The borax added to SET-45 mixtures in our laboratory was manufactured by Fluka Chemical Corp., 255 Oser Avenue, Hauppauge, NY. The chemical name of borax is sodium tetraborate decahydrate ($Na_2B_4O_7 10\ H_2O$). Its actual borax content is greater than 99%.

POLY-N - It is a brand name for 55% water solution of ammonium polyphosphates supplied by Allied Corporation, Philadelphia, PA.

3-Aminopropyl-Triethoxysican - Fluka, Switzerland.

$FeCl_2$, $FeCl_3$ - Fisher Scientific Company, Fair Lawn, NJ, 07610.

3. MECHANICAL TESTS

3.1 Scope

As reported in the Progress Report, four promising rapid hardening materials were tested and after screening, the SET-45 mixtures found to be more promising. Hence the final phase of this report concentrates only on the behavior of SET-45 mixtures at extreme temperatures and with borax combinations. The tests were generally conducted in accordance with pertinent ASTM specifications. Some tests were modified slightly to make them more applicable to the rapid setting nature of the products. Unless otherwise noted, all the

specimens were prepared and tested at ambient laboratory conditions, approximately 75°F (23°C) and 50 percent relative humidity. In addition, specimens were prepared and tests were conducted at high and low temperatures in order to determine the effects of temperature on the setting time and strength development of mortars. The dry mixture, water and curing condition temperatures are given in Table 1. By visualizing the different construction techniques for the emergency repair work, and also considering the shortness of setting times of the SET-45 mortars that have high enough one hour strength, it is obvious that a special, simple but rapid construction method is needed. One such method is the use of a mortar of fluid state instead of plastic or stiff consistency. Not only would this lengthen somewhat the setting time but also it would self level, thus speed up the construction by the elimination of the need for compaction. For this reason the work presented here is focused on mortars of flowing consistency, that is, of flow of approximately 150% as determined by ASTM C 230-80.

3.2 Tests on Fresh Mortars

Mixing

Two mixers were used for mixing the batches: one is of 1/6 cu. ft. and the other is 1/3 cu. ft. capacity bench model. They have three speed gear system with a stainless steel bowl and planetary action. They comply with ASTM C 305.

The mixing procedure used for all batches is, as follows: The mixing water was poured into the bowl, the dry components were premixed and added to the water. These were mixed together first for 30 s at low speed, then for 90 s at medium speed. When borax was

used, it was premixed with the SET-45 cold mixtures.

Flow and Time of Setting Tests

Immediately after mixing, the flow test and time of setting test were performed essentially in accordance with ASTM C 230-80 and C 191-77, respectively.

The standard flows of all fresh mortars were approximately 150%, that is, of flowing consistency. The test results of the setting tests are presented in Figure 1. The water content and borax are expressed as percent by weight of the dry mixture.

Measurement of the Mortar Temperature

Immediately after mixing, temperatures of SET-45 cold and hot mixtures were measured. The mortars were made at ambient laboratory temperature. Then the thermometer was embedded in the center of 3 in x 6 in (76 mm x 152 mm) cylindrical specimens. The purpose was to correlate the heat development to the setting times of SET-45 cold and hot mortars.

The results of the temperature measurements are presented in Table 2 and Figure 2.

3.3 Compressive Strength

Preparation of Specimens

Altogether eight series of tests were performed at different temperatures of dry mixtures, mixing water and curing conditions. Five mixes were made at each temperature series, as follows:

SET-45 cold (SC)

SET-45 hot (SH)

SET-45 cold + hot (SCH)

SET-45 cold + 0.34% borax (SCBA), and

SET-45 cold + 0.7% borax (SCBB).

The various mixes are identified in the tables and figures by the abbreviations in parentheses above. Additional letters attached to these indicate other characteristics of the mixes, as follows:

C cold curing

T hot curing

BA 0.34% borax is present

BB 0.70% borax is present.

For instance, the mix identified as SCBTB is made of SET-45 cold formulation (SC) containing 0.70% borax (BB) and cured at elevated temperature (T).

Disposable waxed card board cylindrical molds of 3 in x 6 in (76 mm x 152 mm) size were used. All specimens were prepared according to ASTM C 192-81 and capped according to ASTM C 617-83 to provide a plane and smooth loading surface. All specimens were removed from the molds approximately 5 to 10 minutes after final set. Compression cylinders were tested according to ASTM C 39-81. Three specimens were tested for each specific age.

First Series

In the first series, all the five mixes were mixed, placed, and air cured at ambient laboratory temperature at 73.4°F ± 3°F (23°C ± 1.7°C) until break. The cylinders were tested at the ages of 1 hour, 3 hours, 24 hours, 7 days, 28 days, and 90 days. The strengths at room temperature are given in Table 3 and Figures 3 through 5.

Other Test Series

In the following series, the mixing and casting were done within 5 minutes at a ambient laboratory temperature and then the specimens were cured at different temperature. Cylinders were tested at the age of 1 hour, 3 hours, and 24 hours.

In the <u>second series</u>, the dry mixtures and water had the ambient laboratory temperature (≈ 73°F) and the specimens were air cured at 32°F (0°C) in a cooled environmental chamber until break. The test results are presented in Table 4 and Figure 6.

In the <u>third series</u>, the dry mixture and water had laboratory temperature and the specimens were air cured at 100°F in an oven until break. The test results are presented in Table 5 and Figure 7.

In the <u>fourth series</u>, only one mix was made. The dry mixture, mixing water, mixing bowl were precooled at 32°F (0°C) and the specimens were air cured at 32°F (0°C) until the break. The test results are presented in Table 6 and Figure 8.

In the <u>fifth series</u>, the dry mixture, mixing water, and mixing bowl were preheated at 100°F (39°C) and the specimens air cured at 100°F (39°C) until break. The test results are presented in Table 7 and Figure 8.

In the <u>sixth</u> and <u>seventh series</u> the dry mixtures had the ambient temperature (73°F) and the water 100°F (39°C). Specimens in the sixth series were air cured at 32°F (0°C) and in the seventh series air cured at 100°F (39°C) until break. The test results are presented in Tables 8 and 9 as well as Figures 9 and 10.

In the <u>eighth series</u>, the dry mixtures had the ambient laboratory temperatures, mixing water had 32°F (0°C) and the specimens were air cured at 100°F (39°C) until break. The test results and temperature details of this mix series are presented in Table 10 and Figure 11. Early age compressive strengths of SET-45 mix series and relationship between one hour compressive strength and time of initial setting are also presented in Figures 12 and 13.

3.4 <u>Flexural Strength</u>

The same five mixes were tested here as for compressive strength. Eighteen 4 in x 4 in x 15 in (101.6 mm x 101.6 mm x 381 mm) beams were prepared in oiled steel molds for each mix essentially in accordance with ASTM C 78. The temperatures of the dry mixtures, mixing water, and curing were at the ambient laboratory temperature (≈73°F). The beams were stripped from the mold 5 to 10 minutes after final set and tested at the age of 1 hour, 3 hours, 24 hours, 7 days, 28 days, and 90 days. Three beams were tested for each specific age. The beams were loaded at the third points of a 12 in span with the loading rate recommended by ASTM C 78-84. The test results are presented in Table 11 and Figures 14 and 15. The relationships between flexural strength and compressive strength for various mixtures are presented in Figures 16 through 21.

3.5 <u>Other Tests on Hardened Mortar</u>

Shear Bond Test

This test was performed essentially according to ASTM C 882. Dummy sections were prepared from portland cement (Type I) concrete (PCC) with the shape and dimensions shown in Figure 22.

After 7 to 10 days hardening the dummy PCC specimen was put in the 3 in x 6 in mold. Then fresh SET-45 mortar was placed on the top of it in the mold in three layers and rodded each layer with 25 storkes. For sake of comparison, the bond of fresh PCC was also tested.

Four bonding mix series were made. They are as follows:

a. Fresh SET-45 cold mortar to hardened PCC, air cured until break. (SCB in Fig. 24)

b. Fresh SET-45 hot mortar to hardened PCC, air cured until break. (SHB in Fig. 24)

c. Fresh PCC to hardened PCC, air cured until break. (OCBR in Fig. 24)

d. Fresh PCC to hardened PCC, moist cured until break. (OCBF)

Tests were carried out at the age of 1 hour, 3 hours, 24 hours, 7 days, 28 days, and 90 days. At each age, two specimens were tested. The bond strength was calculated by dividing the load carried by the specimen at failure by the area of the bonded surface (14.13 in^2). The test results are given in Table 12 and Figures 23 and 24.

Length Changes

Along with the flexural beams, three small beams of 1 in x 1 in x 11 1/4 in (25.4 mm x 25.4 mm x 286 mm) were also cast for each mix to determine the length changes at two different curing conditions. One series of specimens was air cured at ambient temperature and the other two were moist cured at approximately 73°F (23°C) and 100 percent relative humidity. Out of these two latter specimens, one was kept for visual observation, whereas the other for measurement purpose.

All these beams were removed from their molds 5 to 10 minutes after the initial set and the initial length was measured at the age of one hour at ambient temperature. The changes in length of the specimens were periodically measured with an extensometer. The test results are given in Figures 25 and 26.

Specific Gravity, Absorption, and Void Test

For these tests, portions of beams of SET-45 mortars broken in flexure at 90 days were used. The edges were trimmed by using diamond saw cutter. These tests were run in accordance with ASTM C 642-82. The test results are given in Table 13. It was observed that during oven drying very strong ammonia odor was produced by the specimens.

Modulus of Elasticity

Young's Modulus of Elasticity were calculated by using $E = 33\, w^{1.5} \sqrt{f_c'}$ formula. The dry unit weight of the specimens at different ages were calculated by taking the average dry weight of the specimens divided by the volume and expressed in lb/cu. ft. The calculated values are given in Table 14. The relationships between modulus of elasticity and compressive strength are presented in Figures 27 through 32.

4. ANALYSIS AND DISCUSSION OF MECHANICAL TEST RESULTS

The results of mechanical testing in the First Series are summarized in Table 15.

4.1 Flow

For all mix series, mortars of flowing consistency (i.e. 150% flow) were used. The reason was that not only would it lengthen the

setting time, but also it would speed up the construction by the simplification of the repair technique. (Section 7)

It can be seen from the first series in Table 3 that the water required to achieve 150% flow for SET-45 hot mortar is less than that for the SET-45 cold mortar under identical conditions. This is attributed to the presence of borax (boric acid) in the hot mixture. Similar liquifying trend was observed when a small quantity of borax was added to the cold mixture. Consequently, it is not surprising that the water quantities required to achieve flowing consistency for the cold mixture blended with hot mixture, as well as for the cold mixture blended with borax are higher than that for the hot mixture.

The same trend was also observed for all mix series irrespective of the temperature of dry mixture, mixing water, and curing condition.

4.2 Setting Time

The initial and final setting times for SET-45 mortars are shown in Figure 1. These setting times are much shorter than those of the standard portland cements. In the first series, it can be seen that the initial and final setting times of the SET-45 cold mortar is less than 10 minutes at room temperature. SET-45 hot mortar has much longer times of setting even with less water content than the cold mortar. Further, the fifty-fifty blend of cold and hot mixtures as well as cold mixture with borax have in-between setting times. The delay in settings in the hot mixture is due to the coarser MgO phase and the presence of borax. The retarding effect of borax was also observed in SET-45 cold mixture blended with borax. An increase in borax addition increases the set retardation.

In almost all mix series, longer initial setting time was observed for SET-45 hot mixture. Further, it was observed that the slight variations of temperature of the room, dry mixture, and water affect the initial and final setting times as well as the one-hour compressive compressive strength.

4.3 Heat Development During Setting

During the setting process all cements develop heat since the hydration is an exothermic process. The faster the setting, the faster the heat development. Thus the SET-45 cold formula is the one that develops the heat of hydrtion most rapidly and in the largest quantity. (Figure 2) If the time of setting is longer, the heat development is less. The same was observed as in the case of SET-45 hot mixture.

4.4 Compressive Strength

First Series

Relationships between compressive strength and age are shown in Figures 3 through 5. In general, the strengths of SET-45 mortars increase rapidly at early ages (up to 24 hrs) and show a similar trend at late ages (after 28 days). The requirement of at least 2000 psi compressive strength within an hour was achieved for all SET-45 mortars. It can be seen from the figures that, the SET-45 hot mortars exhibit higher strengths than the other SET-45 mortars up to 90 days except for the early age of SET-45 cold mortar blended with 0.34% borax. An independent investigation performed at the University of Texas, Austin also shows that the SET-45 hot mortar exhibits higher

strengths after 24 hrs than the SET-45 cold mortar.*

As expected, the strength increases with age for all mortars similar to conventional portland cements, however, the rate of strength development is greater in the SET-45 mortars. The SET-45 hot mortar achieved 95% of its 24 hours compressive strength within an hour, the other SET-45 mortars showing lesser percentage gains. We have found no adequate uniformity in the SET-45 cold mixture results after three repetitions. As Table 3 shows, the properties change considerably from lot to lot.

It can be seen from Figures 4 and 5 that high strengths were also exhibited by SET-45 cold mixture blended with 0.34% borax. If we compare the test results of SET-45 cold mixture blended with 0.34% borax to that with 0.7% borax, it appears that the higher percent of borax can decisively increase the delay in setting time and reduce the strength at ambient temperature curing.

Other Series

For different combinations of temperatures of ingredients and curing conditions, SET-45 hot mortar exhibits higher early strengths than the other mortars (Figures 6 through 12).

Almost all SET-45 mortars regardless of the combinations of temperatures, surpassed 2000 psi compressive strength at the age of one hour. Among these, the SET-45 hot mortar provided the highest strengths. The University of Texas at Austin findings* also show that higher strength is exhibited by SET-45 hot mortar when cured at

*Research Report No. 311-4 - Laboratory and Field Evaluation of Rapid-Setting Materials used for Repair of Concrete Pavements, July 1984.

higher temperature.

The relationships between one hour compressive strength and the time of initial setting for all mix series are summarized in Figure 13. It appears to show no correlation between the two.

4.5 Flexural Strength

The flexural strength versus age relationship is shown in Figures 14 and 15. In general, higher than 400 psi flexural strength was achieved for all mortars at the age of 1 hour. Like compressive strength, flexural strength shows rapid increases at early as well as late ages (after 28 days) for all SET-45 mixtures. It was also observed that there was a slight drop or no gain in strength for almost all mixes during the intermediate period from 7 through 28 days. The reason for this is not clear.

The SET-45 cold mixture blended with borax (Figure 15) has higher flexural strengths at almost all ages than the other mixtures.

The relationships between flexural and compressive strengths of SET-45 mortars are shown in Figures 16 through 20, and summarized in Figure 21. From Figure 21, the following approximate linear relationship may be established between flexural and compressive strengths:

$$f_{fl} = 0.0357 f_c + 380 \qquad (1)$$

where f_{fl} = flexural strength, psi

f_c = compressive strength, psi.

This equation is valid when the compressive strength of SET-45 mortar is between 2000 and 6000 psi.

4.6 Bond Strength

The bond strength of SET-45 cold and hot mortars are almost the same at early ages (Fig. 23). However, the SET-45 hot mortar exhibited higher bond strengths at later ages. It had also better bond than air and moist cured conventional portland cement concretes.

It is interesting to note that SET-45 hot mortar specimens at the ages of 28 and 90 days as well as the moist cured conventional concrete specimens at the age of 90 days failed in compression rather than in bond (Fig. 24). This indicates excellent bond strengths. After 28 days a rapid increase in bond strength was observed for all mortars. This behavior is similar to that of the compressive and flexural strengths.

4.7 Change in Length

Figures 25 and 26 show changes in length of different unloaded SET-45 mortar bars with age under dry and wet curing respectively. The shrinkage and expansion of SET-45 cold specimens are extensive, that is, 3 to 5 times larger than those of other SET-45 specimens. Knowing that only the hydrated portion of specimens is responsible for change in length, this is not surprising. Due to the fine grading of the MgO phase, the cold formulation must hydrate at a higher rate, thus have the largest length changes. (Fig. 26)

Mechanical tests show that borax is a retarder for setting and early strength which implies that the hydration rate is slowed down. Our X-ray diffraction method was not refined enough to prove or disprove this implication. On the other hand, the low shrinkage measurements of the mortars containing borax imply again that borax

retards the hydration rate causing extended set and reduced early strengths. The large swelling of wet cured SET-45 cold mortar specimens (Fig. 25), however, cannot be explained without the results of X-ray examination. As dicussed in Section 6.4, the unused portion of MgO continues to hydrate in wet cured SET-45 cold specimens, producing an amorphous product the accomodation of which must be responsible for the large swelling during the first two months. Within this period all the MgO is hydrated and also new processes take place. Monohydrate and hexahydrate are decomposed into an amorphous stuff and new crystalline products are formed in the surface layer of the specimens. This may explain the belated shrinkage of the SET-45 cold specimens after two months of wet curing (Fig. 25).

The same trend was observed after the addition of borax to SET-45 cold mixture. After three months no further change in lengths were observed for the wet and dry cured mortars. Especially, the SET-45 hot mortar showed a good volume stability; its length remained unchanged already after 20 days of wet curing.

The SET-45 cold mortar beams kept in the moist room for visual observation did not show any crack, as the earlier ones did presented in our Progress Report (Section 7.3). It is possible that the manufacturer had changed the chemical pattern of SET-45 mixtures recently.

4.8 <u>Specific Gravity, Absorption, Voids, and Modulus of Elasticity</u>

The specific gravity and voids of all the tested SET-45 mortars were practically the same (Table 13). The porosity of the hardened mortars is also practically identical. Hence, only the rate of

hydration influences the strength of the tested specimens of the various mortars.

The relationships between modulus of elasticity and compressive strength of SET-45 mortars are summarized in Figure 32. It is known that the modulus of elasticity is expressed in terms of density and square root of the compressive strength. The graph is drawn however on the basis of calculated data.

The linear relationship as derived from Figure 32 is, as follows:

$$E = 333.3 f_c + 1.77 \times 10^6 \qquad (2)$$

where E = modulus of elasticity, psi

f_c = compressive strength, psi.

This equation is valid when the compressive strength of SET-45 mortars is between 2000 and 9000 psi.

5. PHYSICOCHEMICAL EXAMINATIONS

5.1 Specimen Preparation

Although the mechanical and physicochemical tests were performed concurrently, most of the time not the same mixture or specimens were used for the two tests. With a few exceptions, only the portion of SET-45 was used in physicochemical experiments that passed sieve No. 200. Sieve No. 200 retains most of the sand and the flyash which are parts of SET-45 mixtures; therefore the passing material is referred to as SET-45 cement and SET-45 paste, respectively, when mixed with water. The reason for sieving was that the presence of sand and even flyash interfers with the study of hydration processes where methods like X-ray diffraction and IR

spectoscopy are used. It was assumed that the chemistry of hydration in SET-45 pastes and SET-45 mortars is the closest when the water-cement ratio is kept the same instead of the water-to-solid ratio. For SET-45 mortars water-to-solid ratio of 0.105 by weight corresponds to water-cement ratio of 0.525 by weight. This water-cement ratio was kept constant while working with SET-45 pastes. There is no doubt that even small variations in water content affect the strength of specimens. The variations also affect the hydration processes, of course, but to much lesser extent as it was already described in the Progress Report. Therefore it is reasonable to assume that using water-cement ratio of 0.525 by weight for SET-45 pastes in physicochemical experiments, the results can be applied for the relatively narrow range of water-to-solid ratios of 0.1 through 0.13 which were used in SET-45 mortars for mechanical testing.

The following five mixtures were used for SET-45 pastes all the time, varying only the temperature at mixing an curing:

Name	Identification Mark
SET-45 cold paste	C
SET-45 cold paste + 1.75% borax	C1.75B
SET-45 cold paste + 3.5% borax	C3.5B
SET-45 cold/hot paste (1:1)	CH
SET-45 hot paste	H

The water-cement ratio was 0.525 by weight in each case. The quantities of 1.75% and 3.5% of borax are expressed with respect to the cementitious phase of SET-45. When they are expressed with respect to the total solid, the corresponding quantities are 0.35% and 0.7%

respectively. In a few cases, polyphosphates were added to the mixtures. When doing so, one half of the mixing water was replaced by 55% water solution of ammonium polyphosphate (Poly-N). Specimens of SET-45 pastes were cast in 1" x 2" cylinders. The cylinder molds were not greased before casting in order to avoid any possible chemical interference with the hydration process.

Occasionally, the same specimens were used for mechanical testing and physicochemical examination. In these few cases SET-45 mixture was sieved through sieve No. 50 and the specimens were made with the passing material. 2-in. cubes were cast and were tested for compressive strength at different ages. After the break, the specimens were examined with X-ray diffraction. The water-to-solid ratio of these specimens was 0.29 by weight. Although the sieving is justified for X-ray tests, it is nevertheless a weak point when the results from the mechanical and physicochemical tests are to be compared and correlated. The following two differences should always be considered:

a. SET-45 hydrating paste, without sand and flyash, will produce a higher temperature during hydration than SET-45 hydrating mortar. This difference in the temperature rise can affect the chemistry of hydration.

b. Different sizes of specimens also contribute to some differences, especially when the curing is at low temperatures; namely the evolved heat of hydration escapes faster from small size specimens than from larger specimens and therefore the smaller specimens cool down faster than the larger specimens.

5.2 Additives and Admixtures

In addition to the chemical admixtures described in our Progress Report, many new additives and admixtures were tried. SET-45 phase passing sieve No. 200 was used for these experiments. The additives were solved in the mixing water in different proportions varying from 0.5 g/100 gr of cement up to 25 g/100 gr in some cases. Different inorganic salts were used, such as iron chlorides and sulfates, aluminum phosphates, ammonium phosphates, borax, oxy-sylanes, copper sulfates and chlorides. In short, the effects of chemicals that were expected to interfere either with MgO phase or the phosphate phase of SET-45 mixtures were investigated on the consistency and the time of setting. All the additives, except for borax and ammonium polyphosphate solution, shortened the setting time and stiffened the consistency. Therefore the experiments continued only with borax and polyphosphate solution as described at proper places in this report.

6. RESULTS OF PHYSICOCHEMICAL TESTS

The main hydration characteristics of the tested SET-45 formulations are summarized in Tables 18 through 22.

6.1 Hydration of SET-45 Pastes at 40°C

All five mixtures described in Section 5.1 were tested. Two different temperatures were selected for these experiments, namely 40° and 90°C. Around 40°C are temperatures of the hottest days of a year. However, where concrete surfaces are exposed to the direct sunlight they can reach much higher temperatures than the surrounding air. Therefore experiments at 90°C were also performed.

Materials were preheated to 40°C or 90°C in an oven before mixing. The mixing was performed at room temperature but as fast as possible, not

allowing the materials to cool down appreciably. The mixing was usually completed in 1 to 2 minutes. The paste was cast into preheated 1" x 2" metal cylinders which were then placed back to high temperature and kept their until the time of testing.

X-Ray Diffraction

A general feature, as determined from the X-ray patterns (Figs. 33 through 52), of all five SET-45 combinations cured at elevated temperatures is that the main hydration products are the same as those observed at curing at room temperature and presented in the Progress Report. These are: ammonium magnesium phosphate hexahydrate ($NH_4MgPO_4 \cdot 6H_2O$) and ammonium magnesium phosphate monohydrate ($NH_4MgPO_4 \cdot H_2O$). These will be referred to in this report as hexahydrate and monohydrate, or hexa and mono, respectively. In all the mixtures the monohydrate was present in larger quantities.

A comparison of Figures 33 through 52 to the corresponding graphs obtained with mixes prepared and cured at normal temperature (see: Progress Report, Figs. 21 through 71) reveals again certain similarities and certain differences. The similarities are the following:

a. The hydration is incomplete with respect to MgO in both.

b. The main hydration products are monohydrate and hexahydrate.

The differences are:

c. Monohydrate is favored only in SET-45 cold pastes when cured at normal temperature. In SET-45 hot formulation hexahydrate is the favored hydration product. At elevated temperatures monohydrate is always the main product.

d. Monohydrate/hexahydrate ratio slowly decreases with curing at normal temperature but it increases with curing at elevated temperature. However, it should be emphasized that the curing at $40^{0}C$ went on in the oven where humidity was low. On a hot and humid summer day things may turn out differently. In short, a thermodynamical equilibrium exists between mono- and hexahydrate which seems to be reversible. Both forms normally coexist at ordinary temperatures and humidity. At elevated temperatures in dry air only monohydrate is stable. The equilibrium at elevated temperatures in humid enviroment was not examined. Low temperatures force the equilibrium towards hexahydrate (Section 6.4).

e. A very interesting feature is observed in the X-ray pattern of three of the SET-45 formulations. These are SET-45 cold paste without borax, SET-45 cold paste with 1.75 % borax and SET-45 cold:hot, 1:1 paste. (Figs. 33-40 and 49-52). Namely, a very characteristic diffraction peak of hexahydrate conspicuous at $2\theta = 33.5^{0}$ ($d = 2.67$Å) is either totally missing or reduced in its intensity. (Compare Figs. 41-48 with Figs. 33-40 and 49-52). Its total absence is obvious in X-ray patterns of SET-45 cold paste itself and in SET-45 cold:hot = 1:1 paste (Figs. 33-36 and Figs. 49-52). In SET-45 cold paste with 1.75% borax the peak is always present but reduced in its intensity (Figs. 37 - 40).

If the amount of borax is increased to 3.5% the peak regains it normal intensity. The normal intensity of the peak is also observed in

SET-45 hot paste. In order to interpret this peculiarity, factors that influence or contribute to the intensity of a diffractional peak must be understood. Assuming that the unit cell is unaffected and therefore leaving out the structural factor, such intensity changes can be explained with a preferential orientation of small crystals on the surface or with changes in crystal growth. Preferential orientation is really only an experimental difficulty depending on how the specimen was prepared for X-ray analysis and can be eliminated with redoing the surface. Having done this, it became clear that in our experiments the change in the intensity must be related to the crystal growth of hexahydrate and not to the preferential orientation. A crystal is in different directions generally limited by different planes. It means that when a crystal grows, it grows in different directions in different ways and at different rates. The rate of growth is a temperature dependent quality and changes with temperature in different directions differently, depending on the activation energies and entropies. If these differences are large then one set of planes is already well developed and another has not started forming yet; therefore the corresponding diffraction peak in X-ray pattern is missing or low in its intensity. With other words, a missing peak does not mean necessarily a chemical change. Irregular crystal growth is generally observed, indeed it is very common, at high temperatures.

Assuming that irregular crystal growth is the correct interpretation of the missing diffraction peak in the above mentioned X-ray pattern, the question to be still answered is why this peak is missing only in some samples and not in all of them. For instance, it

is not missing in SET-45 hot paste and SET-45 cold paste with 3.5% borax (Figs. 41-48). It is known from previous tests that one difference between SET-45 cold cement and SET-45 hot cement is that the grain size of MgO in SET-45 hot is much larger than the one in SET-45 cold. The rate of reaction depends, among others, on the grain size: it is faster with fine size and slower with coarser size. At slower rate the irregularities in growth disappear. A very nice example for this is when borax is added to SET-45 cold paste. If only a small amount of borax is added (1.75%) the diffraction peak shows up but its intensity is low (Figs. 37-40). If larger amount of borax is added (3.5%) the intensity of the peak becomes normal (Figs. 41-44). Borax is a known retarder for SET-45 hydration, setting, and hardening (Progress Report, Section 7.2). It slows down the formation of the hydration products which are mostly crystalline and therefore it slows down the crystal growth and, again, the irregularities disappear.

From the practical point of view it is more important to establish how the high rate of hydration reduces the strength. To see this, compressive strength of the corresponding specimens, prepared and used the same way but not sieved, was measured simultaneously with X-ray measurements. Hexahydrate crystal growth pecularities were expected to be reflected in the strength of the specimens and indeed it was observed that specimens with the irregular hexahydrate growth (SCHH and SCBHA in Table 7) show much lower compressive strength than specimens with the normal hexahydrate crystal growth. For example SET-45 hot- and SET-45 cold + 3.5% borax specimens, which produce normal hexahydrate crystals, show higher strengths than the

rest of the specimens which show irregular growth of hexahydrate. On the same basis, the strength of SET-45 cold alone is expected to be lower than the strengths in specimens with borax. Supportive to this speculation are data for specimens which were mixed and cast at normal temperature but were cured at elevated temperature at 39°C (Table 5). They show that SET-45 cold specimens have the lowest strength and that the strength improves with an addition of borax in spite of the fact that borax is a set retarder. Also, borax is a retarder of early strengths for SET-45 mixes prepared and cured at normal temperatures. The fact that borax at elevated temperatures still remains a set retarder but converts at the same time into an accelerator for early strengths seems to be very confusing at first glance but there is an explanation. Namely, if borax is added the setting is retarded, the immediate hydration is retarded, and the corresponding crystal growth is retarded as well. Therefore regular crystals of hexahydrate develop which are beneficial to the strength. On the other hand, without borax the setting time is fast, the immediate hydration is fast and the crystal growth is fast. Therefore the forming crystals are irregular which is less beneficial to the strength than the normal crystals. In other words, one can say that the hydration product at slow rate is of high quality and at fast rate of low quality with respect to the strength. Nevertheless, the typical situation is that a short setting is followed by a relatively high early strength. Similar tendency has been observed with portland cement concrete.

In cases where two differently prepared specimens are compared that form the same quality of hydration product, the typical relation between setting and early strength gain is followed (SHH and SCBHB in Table 7). For example, SET-45 hot paste and SET-45 cold paste with 3.5% borax produce the same quality hydration product, (Figs. 41-48) namely hexahydrate crystals. Accordingly, the normal pattern is observed where faster setting time means also faster early strength gain and vice versa.

Infrared Spectroscopy

Infrared (IR) spectra of the above discussed specimens (Figs. 53-57) are identical in spite of just discussed differences in the X-ray patterns. This is not surprising since if the interpretation of the differences in the X-ray pattern is correct, identical IR patterns are expected. IR spectum is a fingerprint of molecular groups within the unit cell only and does not depend on the growth of crystal. In other words, the IR spectra also suggest that the differences in the X-ray pattern stem from the differences in crystal growth only rather than from structural differences.

It is interesting to compare IR spectra of specimens prepared and cured at 40°C to IR spectra of specimens which were prepared and cured at room temperatures ($\simeq$ 23°C). (Progress Report Figs. 81-84). IR patterns of room temperature curing are characterized by a single band at approximately 1000 cm^{-1} which is in most cases assymetrical having a shoulder. The band was assigned to the hexahydrate and the shoulder to the monohydrate (Progress Report Section 6.7). In IR spectra at 40°C the shoulder develops into a band of

approximately the same intensity as the band assigned to hexahydrate. This means that the amount of monohydrate increases at 40°C, the finding already established from the X-ray patterns.

6.2 Hydration of SET-45 Pastes at 90°C

Not older than 1 day old specimens cured at 90°C were tested considering that the availability of the free water, needed for the hydration to proceed, diminishes fast due to evaporation. Also, results for longer times of curing at so high temperatures are of no practical value knowing that any surface can be exposed to strong sunlight ony for a few hours continuously. The information drawn from this experiment is helpful in better understanding of hydration kinetics and thermodynamics of SET-45 pastes. The hydration kinetics for SET-45 cold and SET-45 hot formulations differ very much at this high temperature (Figs. 58-72). The most important difference, which is probably the reason for all other differences, is that SET-45 cold pastes hydrate totally with respect to MgO phase regardless of the borax content whereas SET-45 hot pastes do not.

The total hydration of MgO phase in SET-45 cold pastes is concluded from the fact that the diffraction peaks at d = 2.1 and 1.49Å (Fig.33), which are the two sharpest peaks for MgO, are nonexistant in Figs. 58-66. An addition of borax does not hinder the MgO hydration but the coarse grain size of MgO does. Namely, the hydration of SET-45 hot paste is far from being completed with respect to MgO (Fig. 67-72). This allows us to have some doubts about the hypothesis that the retarding effect of borax acts through surface protection of MgO grains. This is so, at least at this high curing temperature because if

borax really reduces the effective surface area per weight as coarseness does, the hydration rate of MgO grains in SET-45 cold should slow down with an addition of borax. But it does not, at least at the used high curing temperature.

The hydration products are also very different at high temperatures. Only one product is observed in SET-45 hot pastes. This is the monohydrate. The monohydrate is formed from the beginning and the composition dos not change with curing. Therefore the monohydrate is stable at 90°C. This nicely confirms our previous dicussion on mono- hexa equilibrium (Section 6.1). As reported in our Progress Report, SET-45 hot paste yields mostly hexahydrate at normal temperatures. At 90°C, however, only monohydrate is formed (Figs. 67-69).

The hydration of SET-45 cold pastes takes a new path. This is interesting because at lower temperatures the hydration products are always the same for the both SET-45 formulations, the difference being only in their relative proportion and sometimes in their quality, as for example at 40°C. However, at so high temperature neither hexa- nor monohydrate are formed in SET-45 cold pastes. Phosphates poorer in ammonia and richer in Mg than mono- and hexahydrates are the products as discussed below. Again, an addition of borax does not change the hydration products; and again, this is something that speaks against the protective layer theory at high temperatures. If this theory were true, then the hydration products of SET-45 cold paste with borax ought to be similar to the hydration products in SET-45 hot paste. But this does not happen in SET-45 cold paste (Figs. 61-66).

The only difference between the two SET-45 formulations that may explain the different hydration is the grain size and consequently the amount of the MgO hydrated. MgO phase in SET-45 mixtures is in a much higher amount than needed for the available phosphate. This surplus of MgO does not influence the chemistry of hydration, that is, the formations of mono- and hexahydrates, as long as it does not hydrate which is the usual situation. In this case, the reaction between the MgO and phosphate can be simply written in the following form:

$$MgO + NH_4H_2PO_4\ (aqua) \rightleftarrows MgNH_4PO_4 \cdot nH_2O$$

The reaction can be viewed as a neutralization reaction between basic MgO and acidic $NH_4H_2PO_4$. However, if the excess MgO continues to hydrate after the phosphate is used up, which may happen when the curing temperature is high and the grain size is small, the medium becomes alkaline:

$$MgO + H_2O \rightleftarrows Mg(OH)_2 \rightleftarrows Mg^{2+} + 2OH^-$$

Since ammonium hydroxide is a weak base, the increased alkalinity makes the system unstable with respect to ammonium hydroxide, thus ammonia gas is liberated.

$$NH_4^+ + OH^- \rightleftarrows NH_4OH \rightleftarrows NH_3\ (gas) + H_2O$$

An observed extremely strong smell of ammonia at mixing and curing undoubtedly supports this mechanism. Due to the ammonia losses, phosphates richer in Mg and poorer in NH_3 than $MgMH_4PO_4$ must be formed. The variety of such phosphates is vast but none of them was

positively identified with the registered X-ray patterns*. Nevertheless, the presence of two of them seems to be very probable:

$MgHPO_4 \cdot 3H_2O$ (d = 3.05Å, d = 2.57Å)

and

$Mg_2P_2O_7$ (d = 3.02Å)

$MgHPO_4 \cdot 3H_2O$ may lose some crystal water with curing at high temperatures, and, as a matter of fact, changes in X-ray patterns with the time of curing are observed (Figs. 58 -66).

The conclusion from this experiment is that MgO grain size which controls the rate of hydration, of SET-45 mixtures may change also the hydration path if the grain size is very small and the curing is at high temperature. The following section will show that a very reactive MgO phase change the hydration chemistry also when specimens are cured in humid environment.

6.3 Specimens at Normal Room Temperature but in Humid Environment

It was mentioned in the Progress Report that a few cylinders made of SET-45 cold mixture at high water-to-solid ratios (≥8%) and cured in a fogroom started to crack. (Fig. 20 in the Progress Report) The cracking was observed within the first week of curing and therefore it was considered important to take a closer look at the phenomenon although the durability studies were not a part of the project. Some suggestions regarding the cracking were already made in the Progress Report (Section 7.3) but no further experimental work was done at that

*(Powder Diffraction File, 1981)

time. The reason for this was that the manufacturer discourages (a) the high water-cement ratio used; and (b) wet curing. The offered explanation for cracking was that ammonium magnesium phosphate monohydrate, which is the main hydration product in SET-45 cold paste at normal conditions, recrystalizes into the hexahydrate, thermodynamically more stable form at room temperature, if water is available for the recrystallization process. Due to the higher content of crystal water, the hexahydrate requires a larger volume. This accomodation causes volume increase and possibly cracking.

For the clarification of this harmful phenomenon, new specimens were prepared in such a way as to initiate cracking. Cracking however never occurred. Nevértheless, the detrimental effect of wet curing on SET-45 cold specimens was confirmed by measured reduction in the compressive strength of wet cured specimens as compared to the strength of dry cured specimens (Table 16). Two experiments were performed for the investigation of wet curing on SET-45 cold pastes and mortars.

Wet Cured SET-45 Pastes

In these first experiments 1" x 2" cylinders made of SET-45 cold paste were cast in the way as described in Section 5.1. In addition, a few cylinders were prepared where one half of the mixing water was replaced by an equal volume of 55% water solution of ammonium polyphosphate (Poly-N). [The reasons for using the polyphosphate was that SEM pictures of SET-45 cold pastes with polyphosphate showed a very compact structure of low porosity and therefore were expected to be water resistant (Figs. 73-76)]. One half of the specimens with and

without polyphosphates was cured in a fogroom and the other half in air in the laboratory under dry conditions. The specimens were cured for 1 month and then examined with X-ray, SEM and IR.

The obtained results show that curing dramatically affects specimens of SET-45 cold paste made without polyphosphate (Figs. 77, 78). When X-ray patterns of wet and dry cured specimens are compared, the following observations can be made: both of them show presence of ammonium magnesium monohydrate and hexahydrate in similar proportions. Therefore, the recrystallization did not take place to the anticipated extent. There is, however, a difference in the MgO phase. Namely, a large part of MgO in the dry cured specimen remains unhydrated even after 1 month. On the other hand, the MgO phase hydrates virtually totally in the wet cured specimen. Brucite, $Mg(OH)_2$, is expected to be the product of this additional MgO hydration but the characteristic diffraction peaks are not detected. A broad band of low intensity between $2\theta = 24 - 35^0$ shows up instead. From this peak alone it is impossible to identify the product but the broadness speaks for an ill-crystalline formation. Something similar was observed in SET-45 cold specimens which were cured at freezing temperature (Section 6.4).

SEM picture of 1 month old specimen without polyphosphate also shows distinctive differences between dry and wet curing (Figs. 73-76). Honeycomb formations are very characteristic for the wet cured specimens. New changes take place in the wet-cured specimens when the curing period is 3 months. A layer, 1 or 2 mm thick, is

formed on the surface of specimens. The layer cracks and can be easily peeled off. The thin surface layer and inside portion were examined with X-ray (Figs. 79, 80). The inside loses its crystallinity completely, and no diffraction peaks are detected at all. On the other hand, the outside layer is crystalline, composed of $Mg_3(PO_4)_2 \cdot 8H_2O$ and $(NH_4)H_2PO_4$. A kind of a magnesium phosphate without ammonia and rich in crystal water, as $Mg_3(PO_4)_2 \cdot 8H_2O$, is expected to be formed but presence of $(NH_4)H_2PO_4$ is exactly the ammonium phosphate that is present in dry SET-45 mixtures. Its reappearance in the surface layer can be explained with the following degradation of hexa- or monohydrate:

$$3NH_4MgPO_4 \cdot n\ H_2O \rightleftarrows Mg_3(PO_4)_2 \cdot 8H_2O + (NH_4)H_2PO_4 +$$
$$2NH_3 + (n - 8)\ H_2O$$
$$n = 1 \text{ or } 6$$

Although this process apparently requires wet curing, the specimens can absorb or release some water depending on the mono/hexa ratio at the beginning. Decomposition of mono- and hexahydrate in the inside takes a different path although the end result for both is an amorphous product. This is concluded from the absence of any diffraction peaks. The composition of the amorphous product was not determined.

The significant difference in crystallinity between the layer and the bulk is surprising. One possible explanation for this is that mono- and hexahydrates decompose, either by themselves or in reaction with excess of the hydrated MgO, to the amorphous stuff which later on

begins to crystallize and forms the layer on the surface of the hardened specimen. The overall reaction for such an interpretation may be written as:

$$5NH_4MgPO_6 \cdot nH_2O + MgO + H_2O \rightleftarrows \text{amorphous phase} \rightleftarrows 2Mg_3(PO_6) \cdot$$
$$8H_2O + NH_4H_2PO_6 + (5n - 15)H_2O + 4NH_3 \text{ (gas)}$$
$$n = 1 \text{ or } 6$$

This suggested mechanism accounts for all the obseved features.

SET-45 Cold Paste with Polyphosphate

When X-ray patterns (Figs. 81, 82), SEM pictures (Figs. 73-76) and IR spectra (Figs. 83, 84) of 1 month wet cured specimens of SET-45 cold paste with polyphosphate are compared to dry-cured specimens, no differences are observed at all. This leads to the conclusion that ammonium polyphosphate protects SET-45 pastes against moisture and therefore improves the durability under wet conditions. X-ray patterns reveal that with an addition of polyphosphate the amount of hexahydrate greatly increases, otherwise the patterns are similar to patterns of dry cured SET-45 cold pastes without polyphosphate (Fig. 80). The compositional differences show up in IR spectra (Figs. 83-86). For specimens with polyphosphate a triplet of bands is characteristic and for specimens without polyphosphate a doublet or even a singlet. Knowing that X-ray patterns show a presence of the same crystalline substances, although in different ratios, the differences in IR account for differences in the amorphous part of the pastes. These differences in the amorphous phase should also be responsible for morpholgical differences in SEM

pictures (Figs. 73-76).

Wet-Cured SET-45 Mortars

In these experiments the portion of SET-45 cold mixture was mixed with water that passed through sieve No. 50. This material contained a considerable amount of fine sand. The water-to-solid ratio was 0.29 by weight which corresponds to water-to-solid ratio of 0.11 of unsieved SET-45 mixture. (Progress Report, Fig. 1) Two inch cubes were cast, half of them was cured in the fogroom and the other half in air in the laboratory. The compressive strength of the cubes were tested at different ages. Pieces of the broken cubes were examined with X-ray diffraction method immediately after the break.

The results of this experiment show that a difference exists in the compressive strength between the wet and dry cured specimens already after a few days of curing, the compressive strength of the wet cured specimens being weaker (Table 16). The differences between wet and dry cured specimens in X-ray patterns, however, are not so obvious as they are for the specimens made of SET-45 paste reported in the previous section (Figs. 87-97). This is so because the hydration of the excessive MgO is slowed down at wet curing in specimens containing sand compared to those without sand.

Another interesting difference is noticed when X-ray patterns of SET-45 cold mortars are compared to X-ray patterns of SET-45 cold pastes. Namely, the presence of sand decreases the mono/hexa ratio. The experiments with SET-45 pastes at elevated temperatures offer the explanation (Section 6.1); that is, at higher temperatures the monohydrate is the favored hydration product. Added sand partially

absorbs the evolved heat of hydration and therefore the temperature rise in specimens with sand is lower than in specimens without it. This results in a larger amount of hexahydrate in the hydration product.

In conclusion, the cracking was not reproduced in the wet-cured SET-45 mortars. Note, however, that the mono-hexa recrystallization did not take place either to the expected extent. It is not clear at present why but a probable reason can be variation in composition from one shipment to another; or perhaps the smaller specimen size. In any case, the suggestion that the mono-hexa conversion is responsible for cracking still remains a possibility. It is also possible, however, that an extended hydration of MgO weakens the compressive strength of the wet-cured specimens. This could be also considered the reason for differences in shrinkage (expansion) measurements shown in Fig. 31, and also for the cracking. An addition of ammonium polyphosphate solution slows down the extended hydration of MgO phase, decreases the mono/hexa ratio, and should be therefore a good medicine against cracking regardless which one of the two mechanisms is the correct one. Unfortunately, the polyphosphate solution is also a set and early strength retarder. Borax, a very good set-retarder, impairs the early strength gain less and is therefore preferred when setting time extension is needed.

6.4 Specimens at Low Temperatures

The same five mixes were tested at low temperature as described in Section 5.1. Three test series were prepared under the following conditions:

a. Precooled, cold-cured pastes:

Materials to be mixed were precooled in a freezing room at 0°C where they were also mixed, cast and cured.

b. Preheated, cold-cured pastes:

Materials to be mixed were preheated to 40°C, mixed and cast at room temperature and cured at 0°C.

c. Precooled, warm-cured pastes:

Materials to be mixed were precooled , mixed and cast at 0°C and cured at 40°C in the oven.

The purpose of these tests was to simulate not only the effect of winter and summer climates on the behavior of SET-45 formulations but also whether precooling or preheating of the materials before mixing would counterbalance the harmful effects of the extreme curing temperatures.

Precooled, Cold-Cured Pastes

Samples mixed and cured at 0°C do not set for a long time. Only SET-45 cold paste and SET-45 cold/hot 1:1 paste were stiffened enough after three hours of curing for X-ray patterns to be run. The other samples stiffened enough only after 1 day. SET-45 hot pastes did not set even after 3 days of curing. Interesting, however, is that this difference in setting is not reflected in the X-ray patterns (Figs. 98-116). All of them are very similar regardless of the type of paste and the time of curing. The diffraction peaks of hydration products are of low intensity all the time, meaning that the degree of hydration is low. Ammonium magnesium phosphate hexahydrate is the only crystalline product. Small differences that do exist between the

patterns are too small to be reliably attributed to differences in the hydration products. Complete insensitivity of the X-ray patterns to the time of setting is unexpected because setting portland cement pastes do not show this insensitivity. The virtual equality of X-ray patterns for different SET-45 specimens and for different times of curing is explained with the very slow hydration rate. The rate of hydration is slowed down with low temperature to such an extent, that differences in MgO grain size between the two SET-45 formulations do not matter any longer.

Preheated, Cold-Cured Pastes

Not too many changes are noticed when materials to be mixed were preheated to 40°C and after casting transferred fast to the freezing room. The degree of hydration is still quite low and monohydrate is not formed (Figs. 116-127). This means that the hydration processes are more influenced by the temperature at curing than by the temperature at mixing due to fast cooling of specimens. The rate at which the hydration heat is escaping is therefore very important. An addition of borax clearly lowers the crystallinity of the hydration product indicated by a broad diffraction peak between $2\theta = 24 - 36^{0}$ representing an amorphous stuff (Figs. 122, 123). The main diffraction peaks of hexahydrate are in the same region. Therefore it is possible that the amorphous stuff bears some structural traits of hexahydrate. In portland cement chemistry there is a similar relation between mineral tobermorite and tobermorite gel which is the amorphous product of hydrated cement paste. The ability of borax to lower the crystallinity was already mentioned in the Progress Report.

Precooled, Warm-Cured Pastes

Most interesting are the results from the experiment in which the materials to be mixed were precooled and after casting cured at 40°C (Figs. 128-138). When these X-ray patterns are compared to X-ray patterns of specimens mixed and cured at 40°C (Figs. 33-52), important differences are observed. There the main hydration product was monohydrate whereas in precooled specimens the only product is the hexahydrate. The degree of hydration there was high but remains low in precooled specimens even after longer high temperature curing. The slow forming hydration product at low temperatures apparently tightly seals the MgO grains from further hydration even when the temperature is elevated after the initial hydration. This mechanism is corroborated by comparing Tables 5, 9 and 10 which show strength results for specimens prepared at three different temperatures but all cured at 100°F. Specimens prepared at low temperature show in average the lowest strengths, specimens prepared at normal temperature the highest strengths, and specimens prepared at 100°F medium strengths. Two important conclusions follow from low temperature experiments:

a. Freezing temperature slow down the hydration to a large degree.

b. The temperature of materials before mixing is very important. If their temperature is low the hydration will be slow even at high curing temperatures.

As discussed in Section 5.1, the results can be influenced by the specimen size. This is especially true when specimens are cured at

low temperature. While large specimens with sand, used in mechanical testing, have a large heat capacity and therefore cool down slowly, small specimens without sand cool down in no time. This difference is nicely reflected, for example, in setting times. Setting times of large specimens are mainly determined by the temperature of the materials at mixing and less by the temperature of curing. The time of setting for small specimens depends on the other hand, on the temperature of curing and less on the temperature at mixing. In other words, small specimens cured at low temperatures set much later than large specimens. In some cases it took over three days before the setting of a small specimen occurred at 0°C temperature. Therefore it is expected that the hydration processes in small specimens at low temperatures are delayed or even different than the processes in large specimens. In practice this means that a few centimeter thick layer of SET-45 mortar will have different properties than a large block of SET-45 mortar when cast in cold weather. The physicochemical tests approximate the cooling situation in a thin layer.

7. COMPARISON OF THE TESTED MATERIALS

Based on the results obtained, the advantages and disadvantages of each SET-45 mixture under investigation can be summarized as follows:

a. SET-45 hot mortar required the least amount of mixing water to achieve flowing consistency that is approximately 150% flow measured according to ASTM C230-80. The water required for SET-45 cold blended with borax is less than that for SET-45

cold mortar but greater than that for SET-45 hot mortar (Table 3).

b. SET-45 hot mixture produces the best times of initial setting at every elevated temperatures. For instance the setting time at room temperature is longer than half hour which is the longest one tested. SET-45 cold mixture has initial setting in less than 10 min. which is the shortest one. The setting times of the other tested mixture fall in between these two extremes. (Fig. 1).

c. SET-45 hot mortar develops less heat during setting and hardening than SET-45 cold mortar (Fig. 2).

d. SET-45 hot mortar has higher compressive strengths than the other SET-45 mortars at ambient temperature. The next highest strength is produced by SET-45 cold mortar blended with 0.34% borax (Figs. 3 through 5).

e. Under different combinations of temperatures of ingredients and curing conditions, SET-45 hot mixture usually exhibits the highest strength.

f. At elevated curing conditions, the second highest strength was obtained with SET-45 cold mortar blended with 0.7% borax. This, in turn, has higher strengths than the SET-45 cold blended with 0.34% borax (Tables 5 and 7) (Figs. 7 and 8).

g. Based on the objectives described in Section 1.2, it is possible to rank the SET-45 mixtures according to their perceived applicability for emergency repair. This includes the adequacy of the setting time and early strength at different

combinations of temperatures. For instance, SET-45 cold mixture exhibits the highest one hour strength at room temperature. Nevertheless it doesn't receive the top rating because its setting time is less than 10 min. The ranks of SET-45 mixtures are presented in Table 17 where 1 represents the highest rank and 3 the lowest.

h. In general, SET-45 cold mortar blended with 0.34% borax exhibits higher flexural strength than the other mortars. The flexural strength of SET-45 hot mortar was practically the same as that of the other mortars (Figs. 14 and 15).

i. SET-45 hot mortar has better bond strength than the others, including portland cement concrete (Figs. 23 and 24).

j. There is almost or no shrinkage or expansion at early ages for SET-45 mortars. At later ages, that is at 100 days the maximum drying shrinkage for SET-45 hot mortar was 0.047 percent, while the max drying shrinkage for SET-45 cold mortar was 0.111 percent. The most stable was the SET-45 cold mortar blended with borax for both types of curing: at 100 days dry curing the shrinkage was 0.027 percent, and at 100 days moist curing, the expansion was 0.0044 percent (Figs. 25 and 26). Note that both the SET-45 hot and SET-45 cold mortars blended with borax exhibit less change in length than that for portland cement mortar.

8. CONSTRUCTION TECHNIQUE

8.1 General Guidelines

The following suggestions are offered for use of SET-45 materials in the emergency repair works:

a. The mixer should be efficient, that is, should blend uniformly and quickly.

b. All materials should be close to the repair in sufficient quantities.

c. Mixing for SET-45 mixtures should be short and done near the damaged spot because of the rapid setting nature.

d. SET-45 dry mixture and mixing water should be prevented from reaching hot or freezing temperatures when used. In other words, the temperature of the mortar should be close to room temperature at the time of application.

e. Plenty of water should be on hand for cleaning. It is also advisable to keep borax powder on hand. In case that the mortar cannot be poured out of the mixer before the setting starts, the borax should be poured into the mixer and mix it with the mortar.

8.2 Major Steps of Construction

A construction method for emergency repair of runways must be quick, simple and foolproof, using durable equipment. The application of mortar of flowing consistency is a major step in this direction.

In case when the damage is in the form of large crater(s), the major steps of a construction technique may be, as follows:

a. Push the broken pieces of the damaged pavement as well as those of the base course and subgrade back to the crater.

b. If this does not fill up the crater completely, add enough crushed stone to it.

c. Compact this loose mass, for instance by a roller.

d. Pour a rapid-hardening liquid cementing material on the top until the voids in the compacted but still granular mass are filled up (similarily to the "prepacked" technique) with or without vibration.

e. Finish the surface.

Another possible case is when the damage is essentially surface damage, similar to potholes. In this case a possible repair technique would consist of the following steps:

a. Remove the loose pieces from the broken surfaces of the runway for instance by compressed air or water jet or jack hammer.

b. Place rapid hardening mortar to the "potholes." Use fluid mortar because it is self leveling and requires no, or very little compaction.

c. Finish the surface.

d. If the depth of pothole is more than, say, 4", fill up with aggregate and pour SET-45 mortar on the top of it and finish it.

9. CONCLUSIONS AND RECOMMENDATIONS

Test results of the mechanical and physicochemical examinations have led to the conclusion that SET-45 hot mortar of flowing consistency appears to be the most suitable for emergency repair of

runways under all weather conditions.

The reasons for recommending SET-45 hot mortar are, as follows:

a. It requires the least amount of water to achieve flowing consistency.

b. It exhibits the longest setting times without any extra admixture.

c. It achieves the highest or almost the highest early and late compressive strengths. For instance, its one-hour strength is higher than 2500 psi at room temperature even with flowing consistency.

d. It shows adequate flexural strength.

e. It provides highest bond strength to old concrete.

f. It exhibits good volume stability.

Based on the physicochemical tests, the following general conclusions are drawn:

a. Chemically active part of SET-45 mixtures consists of MgO grains and $NH_4H_2PO_4$ solid particles. Boric acid or borax may or may not be present. The inactive part consists of sand and flyash. SET-45 mixtures are manufactured in cold- and hot-weather formulations. The hot weather formulation contains coarser MgO phase than the cold weather formulation and a small addition of boric acid for longer setting times.

b. The hydration is always complete and very fast with respect to $NH_4H_2PO_4$. However, it is incomplete with respect to MgO phase

most of the time. (Fig. 33) MgO phase hydrates totally only in SET-45 cold weather pastes either when the hydration takes place at very high temperatures (~90°C) or when it is cured at high humidity for at least a month. (Figs. 61 and 78)

c. Ammonium magnesium orthophosphate monohydrate and ammonium magnesium orthophosphate hexahydrate are the crystalline parts of the hydration product. These are referred to as "mono" and "hexa", respectively. Only the hydration of SET-45 cold weather paste at very high temperatures may result in some other crystalline products. (Fig. 78)

d. The mono-hexa ratio and the quality of the crystalline phase of the hydration product depend on the rate of hydration. The monohydrate is the main crystalline product when the hydration is rapid, that is, its amount increases with the fineness of MgO phase and with temperature. Conversely, the amount of hexahydrate decreases with the same parameters. The quality of hexahydrate crystals is also affected by temperature because they may grow irregularly when the hydration rate is high.

e. During the curing period the mono-hexa ratio may change. It may increase with high temperatures and dry environment, or it may decrease with low temperatures and humid environment. Perhaps not coincidentally, the total volume of the crystalline phase also decreases with low temperature and high humidity.

f. Some of these chemical changes are harmful. These are described under b, c, d and e above and are characteristic especially of SET-45 cold weather pastes. The harm is that they

cause extensive expansion in wet cured SET-45 cold weather specimens and perhaps not independently reduce the strength. (Figs. 25, 26) This can also initiate cracking. An addition of borax reduces the harmful expansion but the mechanism for its action is not known. As it was established, borax does not change the mono-hexa ratio but it changes the morphology, that is, lowers the crystallinity and perhaps lowers the activity of the MgO phase.

g. In contrast to SET-45 cold formulation, SET-45 hot pastes and blends of SET-45 cold and hot formulations do not suffer any large chemical changes during curing. Thus, they are considered stable and more durable than SET-45 cold pastes.

h. High hydration rate is not recommended because irregular hexahydrate crystals may be formed and the MgO phase may hydrate to an excessive extent. Both effects lower the strength. Therefore mixes with coarse MgO phase and with borax, both having retarding effect, perform better than mixes with fine MgO phase and without borax.

i. A very low hydration rate, which is the case at low temperature, is also not recommended. The hydration product, low in crystallinity apparently seals off the MgO phase thus preventing it from further hydration. Later curing at higher temperatures therefore does not reverse the ill-effect completely.

j. The above statements, strictly speaking, refer to SET-45 pastes. Sand and flyash, which are present in marketed SET-45 mixures are inert, nevertheless they influence the hydration through

the absorption of heat of hydration.

From the test results and conclusions, the following is recommended:

a. Use SET-45 hot mixture as the first choice in all weather conditions in flowing consistency.

b. If this is not possible, use SET-45 cold mixture blended with 0.34% borax, except during hot weather when 0.7% borax should be used in the mixture. The addition of borax to SET-45 cold mixture extends the setting time without any detrimental effects on strengths.

c. The temperature of the mortar should be close to room temperature at the time of application.

d. Use the simplest and quickest construction method including mixing.

TABLE 1 - Temperature Details of SET-45 Mix Series

Mix Series Number	Temperature, °F*				Number of Mixes in each series
	SET-45 dry Mixture	Water	Mixing Bowl	Air Curing	
1	73	73	73	73	5
2	73	73	73	32	5
3	73	73	73	100	5
4	32	32	32	32	1
5	100	100	100	100	5
6	73	120	73	32	4
7	73	100	73	100	5
8	73	32	73	100	5

*Temperature fluctuation ± 2 °F

°C = (°F - 32)/1.8

TABLE 2 - Temperatures of SET-45 Mortars During Setting at Ambient Temperature

Time Min.	Temperature, °F		Time Min.	Temperature, °F	
	SET-45 Cold	SET-45 Hot		SET-45 Cold	SET-45 Hot
0	75	75	27	**	163
1	75	75	28	**	167
2	77	77	29	**	172
3	79	77	30	176	176
4	81	79	31	**	176
5	83	81	32	**	176
6	86	83	33	**	172*
7	90	**	34	**	**
8	93	84	35	165	**
9	97	**	40	158	167
10	100*	86	45	149	***
11	106	**	50	142	162
12	109	88	55	135	**
13	122	**	60	127	149
14	133	95	65	118	**
15	149	98	70	113	**
16	185	100	90	75	**
17	190	102	120	**	75
18	190	104			
19	190	106			
20	190	108			
25	185	138			

*Approximate final set time.

**Temperatures were not measured.

°C = (°F - 32)/1.8

TABLE 3 - First Series: Setting and Compressive Strength of SET-45 Mortars

All the specimens were air cured at ambient temperature.

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi					
			Initial	Final	1 hr	3 hrs	24 hrs	7 d	28 d	90 d
SC(1)	11	-	7 min 30 sec	9 min 30 sec	2580	2810	2920	4020	4690	7230
SC(2)**	11	-	7 min 30 sec	9 min 30 sec	3290	3030	3640		6040	
SC(3)**	11	-	13 min	14 min	2490	2730	2560			
SH	10	-	34 min 30 sec	37 min 15 sec	2930	3000	3100	5020	6630	8620
SCH	11	-	16 min	20 min	2840	3080	3220	3770	4580	6870
SCBA	10.5	0.34	11 min 30 sec	13 min 30 sec	3150	3160	3840	4360	4940	7920
SCBB	10.5	0.70	24 min	27 min	1920	2140	2850	3340	3830	6170

*Percent by weight of the dry mixture.

**Repetition

145 psi = 1 MPa

TABLE 4 - Second Series: Properties of SET-45 Mortars Air Cured At 32°F (0°C) I

The dry mixture and mixing water had ambient temperature.

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCC	11	-	10 min	12 min	2700	2800	2840
SHC	10	-	40 min	44 min	2690	3690	3850
SCHC	11	-	21 min	25 min	2240	2660	2670
SCBCA	10.5	0.34	31 min	33 min	2020	2860	2990
SCBCB	10.5	0.70	45 min	50 min	710+	1130	2990 3570++

*Percent by weight of the dry mixture

+Since the specimens were too weak for one-hour test, this value was obtained at 2 hrs.

++15 day compressive strength

TABLE 5 - Third Series: Properties of SET-45 Mortars Air Cured at 100°F (39°C) I

The SET-45 dry mixture and mixing water had ambient temperature.

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCT	11	-	10 min	11 min	2370	2530	2740
SHT	10	-	17 min 30 sec	19 min	3240	3600	4120
SCHT	11	-	16 min 15 sec	19 min	2600	2860	3340
SCBTA	10.5	0.34	17 min 30 sec	20 min 15 sec	2250	2460	2910
SCBTB	10.5	0.70	24 min 30 sec	26 min 30 sec	2300	2820	3190

*Percent by weight of the admixture

TABLE 6 - Fourth Series: Properties of SET-45 Cold Mortars
Air Cured at 32°F (0°C) II

The dry mixture, mixing water, and mixing bowl were precooled at 32°F (0°C).

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCCA	12	-	1 hr 10 min	1 hr 45 min	**	2090	3200

*Percent by weight of the dry mixture

**The specimens were too weak for the one-hour test

TABLE 7 - Fifth Series: Properties of SET-45 Mortars Air Cured at 100°F (39°C) II

The dry mixture, water and mixing bowl were preheated at 100°F (39°C).

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCH	13	-	1 min	1 min 10 sec	**	**	**
SHH	12	-	4 min	6 min	2390	2660	2520
SCHH	13	-	3 min	3 min 30 sec	1820	1910	1900
SCBHA	13	0.34	2 min	2 min 30 sec	1840	2000	2270
SCBHB	13	0.70	3 min	3 min 30 sec	2420	2520	2560

*Percent by weight of the dry mixture

**Cylinders could not be cast because of rapid setting

TABLE 8 – Sixth Series: Properties of SET-45 Mortars Air Cured at 32°F (0°C) III

The dry mixtures had room temperature and the mixing water 120°F (49°C).

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCCH	11	-	3 min	3 min	1820	1910	2120
SHCH	10	-	16 min	18 min	3470	3770	4050
SCHCH	11	-	7 min 30 sec	10 min	3150	3060	3290
SCBCHB	10.5	0.7	12 min 10 sec	16 min 45 sec	3070	3240	3610

*Percent by weight of the dry mixture

TABLE 9 - Seventh Series: Properties of SET-45 Mortars
Air Cured at 100°F (39°C) III

The dry mixtures had ambient temperature and the mixing water 100°F (39°C).

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCHH	11	-	5 min 14 sec	6 min 25 sec	3160	3460	3870
SHHH	10	-	18 min	20 min	3090	3250	4190
SCHHH	11	-	11 min 5 sec	12 min 55 sec	2230	2280	2480
SCBHHA	10.5	0.34	9 min 20 sec	11 min 48 sec	2490	2460	2760
SCBHHB	10.5	0.70	13 min 47 sec	15 min 12 sec	1920	1990	2420

*Percent by weight of the dry mixture

TABLE 10 - Eighth Series: Properties of SET-45 Mortars Air Cured at 100°F (39°C) IV

The dry mixtures had ambient temperature and the mixing water 32°F (0°C).

Mix Designation	Water* %	Admixture* Borax %	Setting Time		Compressive Strength, psi		
			Initial	Final	1 hr	3 hrs	24 hrs
SCHC	11	-	28 min 50 sec	33 min 44 sec	1570	1900	2130
SHHC	10	-	49 min 52 sec	55 min 25 sec	**	2230	2800
SCHHC	11	-	43 min 44 sec	49 min 55 sec	**	1650	2360
SCBHCA	10.5	0.34	29 min 50 sec	34 min 5 sec	1960	2100	2750
SCBHCB	10.5	0.70	38 min 12 sec	42 min 54 sec	**	2060	2460

*Percent by weight fo the dry mixture

**The specimens were too weak for the one-hour test

TABLE 11 - First Series: Flexural Strength of SET-45 Mortars.

The specimens were air cured at ambient temperature.

Mix Designation	Water* %	Admixture Borax* %	Flexural Strength, psi**					
			1 hr	3 hrs	24 hrs	7 d	28 d	90 d
SC	11	-	435	480	490	505	530	780
SH	10	-	440	490	490	575	525	605
SCH	11	-	460	465	480	485	475	600
SCBA	10.5	0.34	470	500	565	560	565	660
SCBB	10.5	0.70	455	490	495	540	525	670

*Percent by weight of the dry mixture

**Third point loading

TABLE 12 - Bond Strength of SET-45 Cold, Hot Mortars and Conventional Concrete

The strength specimens were 3" x 6" cylinders.

Mix Designation	Water*	Bond Strength, psi						Remarks
	%	1 hr	3 hrs	24 hrs	7 d	28 d	90 d	
SCB	11	1080	1100	1280	1545	1590	2135	Air cured
SHB	10	1080	1150	1270	1610	>1955 NBF	>2165 NBF	Air cured
OCBR	0.45+	-	-	550	1040	1540	1570	Air cured
OCBF	0.45+	-	-	870	1620	1730	>2130 NBF	Moist cured

*Percent by weight of the dry mixture

+Water/cement ratio

NBF - No Bond Failure

TABLE 13 - Specific Gravity, Absorption, and Voids of SET-45 Mortars

Serial No.	Test Details	SET-45 Cold	SET-45 Hot	SET-45 Cold + Hot	SET-45 Cold + 0.34% borax	SET-45 Cold + 0.7% borax
1	Absorption after immersion, %	8.766	7.830	8.720	8.997	9.008
2	Absorption after immersion and boiling, %	8.961	8.870	9.633	9.810	9.134
3	Bulk specific gravity, dry	1.970	2.045	2.026	1.988	1.985
4	Bulk specific gravity after immersion	2.151	2.211	2.203	2.166	2.163
5	Bulk specific gravity after immersion and boiling	2.147	2.227	2.222	2.183	2.166
6	Apparent specific gravity	2.393	2.498	2.518	2.469	2.424
7	Volume of permeable pore space (voids), %	17.67	18.13	19.54	19.48	18.12

TABLE 14 – First Series: Modulus of Elasticity of SET-45 Mortars

All the specimens were air cured at ambient temperature.

Mix Designation	Water* %	Admixtures Borax* %	Modulus of Elasticity (10^{-6}psi)					
			1 hr	3 hrs	24 hrs	7 d	28 d	90 d
SC	11	–	2.69 (136.96)	2.79 (136.50)	2.81 (135.43)	3.16 (131.73)	3.82 (130.32)	4.12 (129.27)
SH	10	–	2.77 (133.85)	2.77 (132.88)	2.83 (133.30)	3.57 (132.63)	4.13 (133.10)	4.66 (132.33)
SCH	11	–	2.71 (133.34)	2.77 (131.85)	2.86 (132.56)	2.97 (129.04)	3.26 (128.80)	3.96 (127.96)
SCBA	10.5	0.34	2.84 (133.12)	2.83 (132.50)	3.16 (133.63)	3.17 (128.52)	3.34 (127.60)	4.21 (127.19)
SCBB	10.5	0.70	2.35 (138.29)	2.47 (137.87)	2.84 (137.57)	3.06 (137.14)	3.25 (136.37)	4.06 (134.96)

*Percent by weight of the dry mixture

( ) Indicates unit weight of the specimen, lb/cu. ft.

TABLE 15 - First Series: Summary of Mechanical Test Results

Age of Testing / Mix Details	SET-45 Cold	SET-45 Hot	SET-45 Cold + Hot Borax	SET-45 Cold + 0.34% Borax	SET-45 Cold + 0.7%
Setting Time					
Initial	7 min. 30 sec.	34 min. 30 sec.	16 min.	11min. 30 sec.	24 min.
Final	9 min.	37 min. 15 sec.	20 min.	13 min. 30 sec.	27 min.
Compressive Strength, psi					
1 Hour	2580	2930	2840	3150	1920
3 Hours	2810	3000	3080	3160	2140
24 Hours	2920	3100	3220	3840	2850
7 days	4020	5080	3770	4360	3340
28 days	4690	6630	4580	4940	3830
90 days	7230	8620	6870	7920	6170
Flexural Strength, psi					
1 Hour	435	440	460	470	455
3 Hours	480	490	465	500	490
24 Hours	490	490	480	565	495
7 days	505	575	485	560	540
28 days	530	525	475	565	525
90 days	780	605	600	660	670
Shear Bond, psi (1)					
1 Hour	1080	1080	-	-	
3 Hours	1100	1150	-	-	-
24 Hours	1280	1270	-	-	-
7 days	1545	1610	-	-	-
28 days	1590	>1955(B)	-	-	-
90 days	2135	>2165(B)	-	-	-

Table 15 (Continued)

Mix Details / Age at Testing	SET-45 Cold	SET-45 Hot	SET-45 Cold + Hot	SET-45 Cold + 0.34% Borax	SET-45 Cold + 0.7% Borax
			Length Change, %		
Dry curing					
1 day	0	0	0	0	0
3 days	0	0	0	0	0
7 days	0	0	0	0	0
20 days	-0.018	0	0	0	0
28 days	-0.027	-0.008	0	0	0
60 days	-0.108	-0.040	-0.018	-0.018	-0.013
90 days	-0.111	-0.047	-0.049	-0.035	-0.027
100 days	-0.111	-0.047	-0.049	-0.035	-0.027
Moist curing					
1 day	0	0	0	0	0
3 days	0	0	0	0	0
7 days	0.009	0	0	0	0
20 days	0.027	0	0	0	0
28 days	0.040	0.018	0.0044	0.0044	0.0044
60 days	0.044	0.018	0.0044	0.0044	0.0044
90 days	0.031	0.018	-0.0044	0	0
100 days	0.031	0.018	-0.0044	0	0

TABLE 16 - Cube Strength of SET-45 Cold Mortar** under Moist and Dry Curing.

Mix Designation	Type of Curing	Water* %	Compressive Strength, psi				
			1 hr	3 hrs	24 hrs	7 d	28 d
SCS	Moist	11	810	910	880	1020	1260
	Dry	11	810	860	825	1650	1880

*Percent by weight of the unsieved dry mixture

**Dry mixture passed sieve #50

TABLE 17 - Ranking of SET-45 Mixtures

Rank 1 is the best.
The term "Hot" refers to SET-45 hot-weather formulation, and "Cold" to cold-weather formulation.

Mix Series	Temperatures of ingredients and curing	Ranking	Remarks
1	Simulation of normal weather conditions: Ingredients, mixing bowl and curing had room temperature.	1. Hot 2. Cold + 0.34% borax 3. Cold + Hot	
2	Simulation of Winter conditions I: Ingredients and mixing bowl had room temperature but the specimens were cured at 32°F.	1. Hot 2. Cold + 0.34% borax 3. Cold + Hot	
3	Simulation of Summer conditions I: Ingredients and mixing bowl had room temperature but the specimens were cured at 100°F.	1. Hot 2. Cold + 0.7% borax 3. Cold + Hot	
4	Simulation of Winter conditions II: Ingredients, mixing bowl were precooled at 32°F and the specimens were air cured at 32°F.	1. Cold	Early strengths are low. The use of ingredients of room temperature is recommended (See Mix Series 2).
5	Simulation of Summer conditions II: Ingredients, mixing bowl were preheated and the specimens were air cured at 100°F.	1. Hot 2. Cold + 0.7% borax 3. Cold + 0.34% borax	The setting time is too short. The use of ingredients of room temperature or cooled water is recommended. (See Mix Series 3 and 8).

Table 17 - Continued

Mix Series	Temperatures of ingredients and curing	Ranking	Remarks
6	<u>Simulation of Winter conditions III</u>: SET-45 dry mixture and mixing bowl had room temperature and water had 120°F but the specimens were cured at 32°F.	1. Hot 2. Cold + 0.34% borax 3. Cold + Hot	
7	<u>Simulation of Summer conditions III</u>: Ingredients and mixing bowl had room temperature and water had 100°F but the specimens were cured at 100°F	1. Hot 2. Cold + 0.34% borax 3. Cold + Hot	
8	<u>Simulation of Summer conditions IV</u>: Ingredients and mixing bowl had room temperature and water had 32°F but the specimens were cured at 100°F	1. Hot 2. Cold + 0.34% borax 3. Cold	

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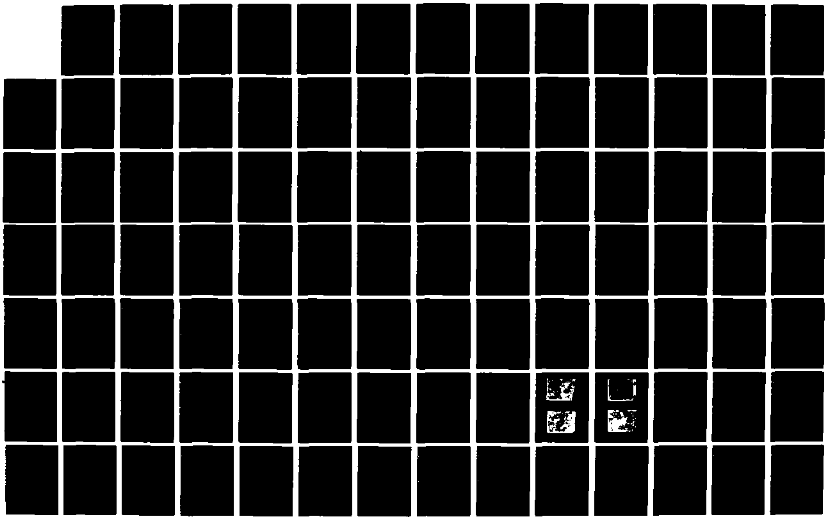

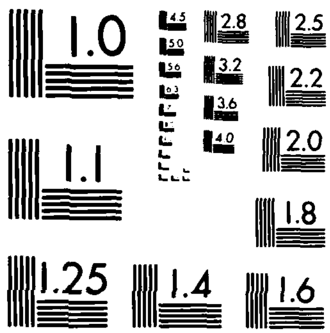

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TABLE 18 - Hydration Characteristics of SET-45 Cold Pastes

Characteristics	Curing Temperature 30°F	77°F Dry	77°F Wet Short Period	77°F Wet Long Period	100°F	200°F
Unhydrated MgO	Present	Present	Present	Absent	Present	Absent
Monohydrate	Absent	Present	Present	Absent	Present	Absent
Hexahydrate	Present	Present	Present	Absent	Present	Absent
Mono/Hexa	<<1	>1	>1	NA	>1	NA
Degree of Hydration	low	medium	medium	high	high	high

TABLE 19 - Hydration Characteristics of SET-45 Cold Pastes with 1.75% Borax

Characteristics	Curing Temperature 30°F	77°F	100°F	200°F
Unhydrated MgO	Present	Present	Present	Absent
Monohydrate	Absent	Present	Present	Absent
Hexahydrate	Present	Present but irregular	Present	Absent
Mono/Hexa	<<1	>1	>1 increases with time	NA
Rate of Hydration	low	medium	high	high

TABLE 20 - Hydration Characteristics of SET-45 Cold Pastes with 3.5% Borax

Characteristics	Curing Temperature 30°F	77°F	100°F	200°F
Hydrated MgO	Present	Present	Present	Absent
Monohydrate	Absent	Present	Present	Absent
Hexahydrate	Present but not always. Amorphous *stuff forms.*	Present	Present	Absent
Mono/Hexa	N/A	>1	>1 increases with time	N/A
Degree of Hydration	low	medium	high	high

TABLE 21- Hydration Characteristics of SET-45 Hot Pastes

Characteristics	Curing Temperature 30°F	77°F	100°F	200°F
Unhydrated MgO	Present	Present	Present	Present
Monohydrate	Absent	Present	Present	Present
Hexahydrate	Present	Present	Present	Absent
Mono/Hexa	<<1	<1 decreases with time	>1 increases with time	N/A
Degree of Hydration	low	medium	high	high

TABLE 22- Hydration Characteristics of Pastes of SET-45 Cold + Hot Combinations

Characteristics	Curing Temperature 30°F	77°F	100°F	200°F
Unhydrated MgO	Present	Present	Present	Present
Monohydrate	Absent	Present	Present	Present
Hexahydrate	Present	Present	Present but irregular	-
Mono/Hexa	<<1	<1	>1	NA
Degree of Hydration	low	medium	high	high

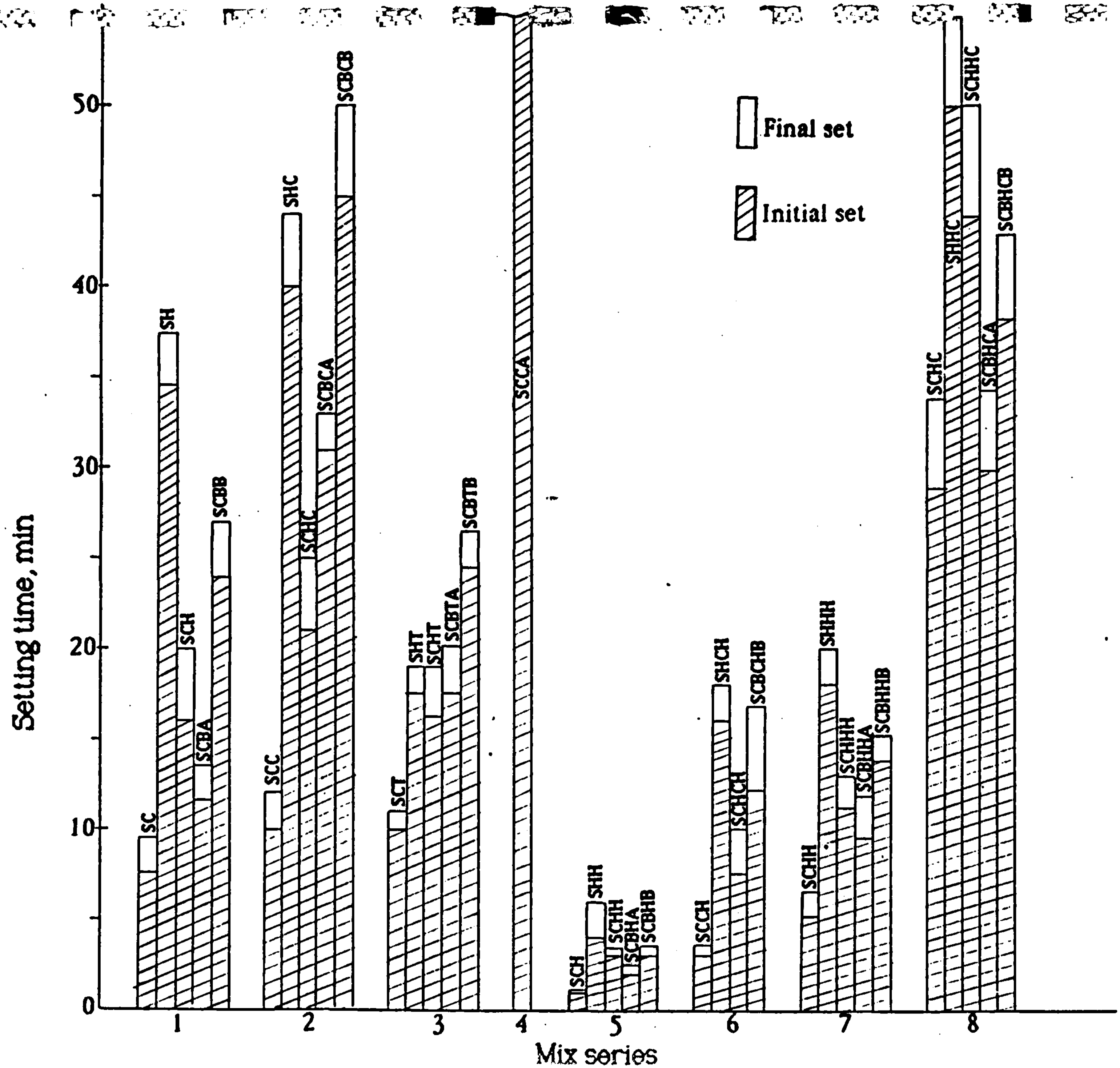


Fig.1 - Setting time of SET-45 mortars

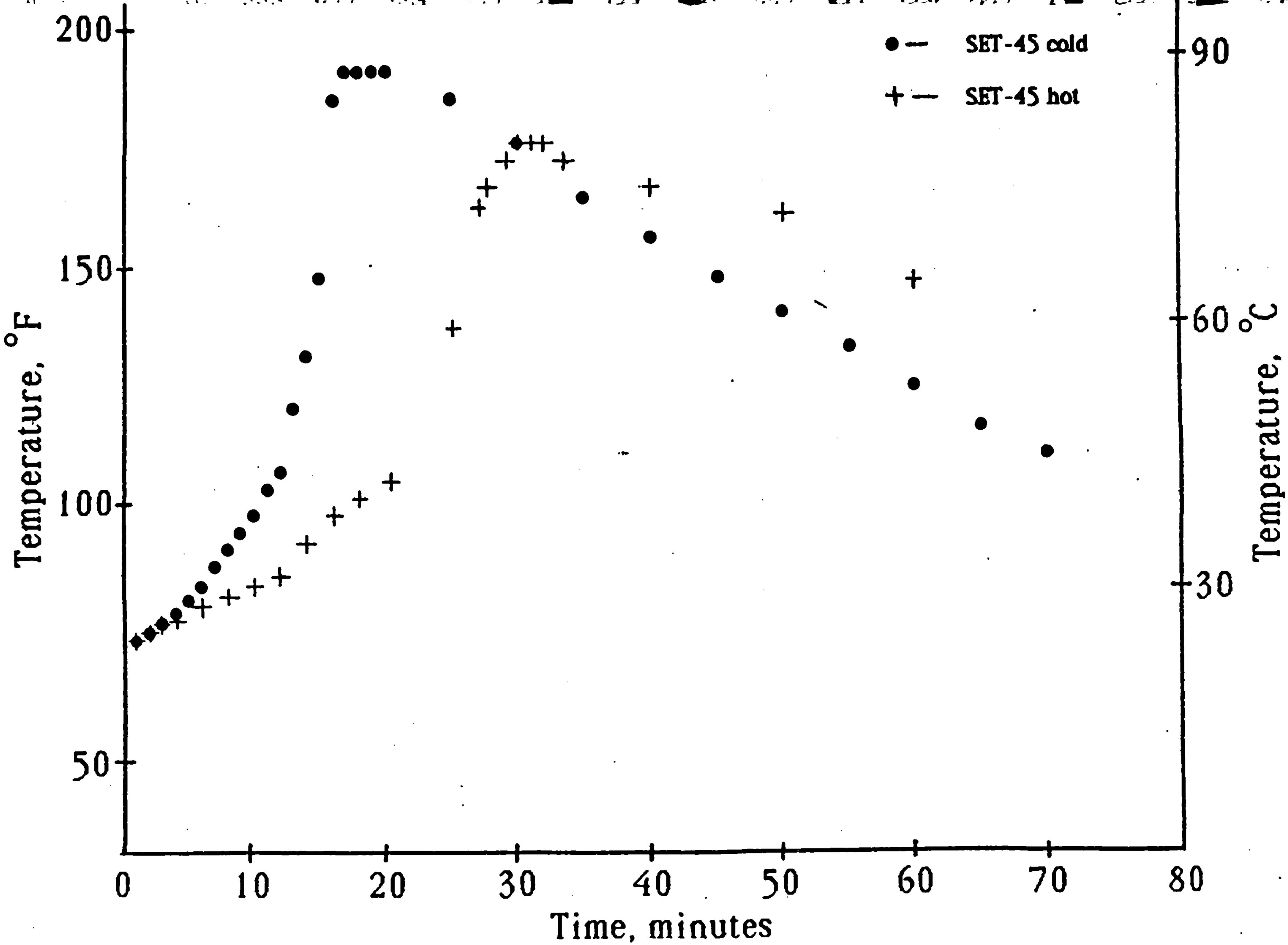


Fig. 2 - Temperatures of SET-45 mortars during setting at ambient temperature and 50% relative humidity

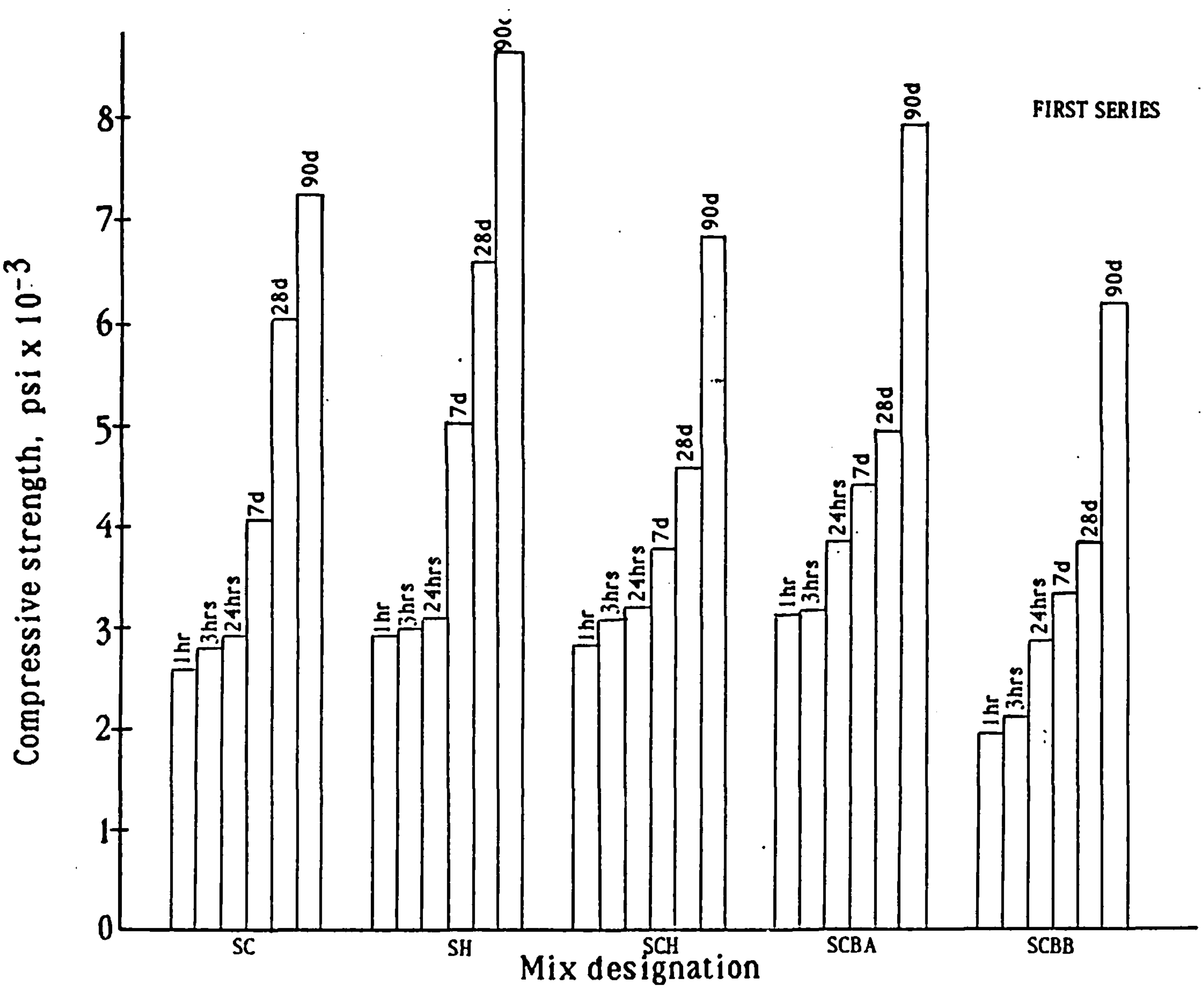


Fig.3 - Compressive strengths of SET-45 mortars at ambient temperature

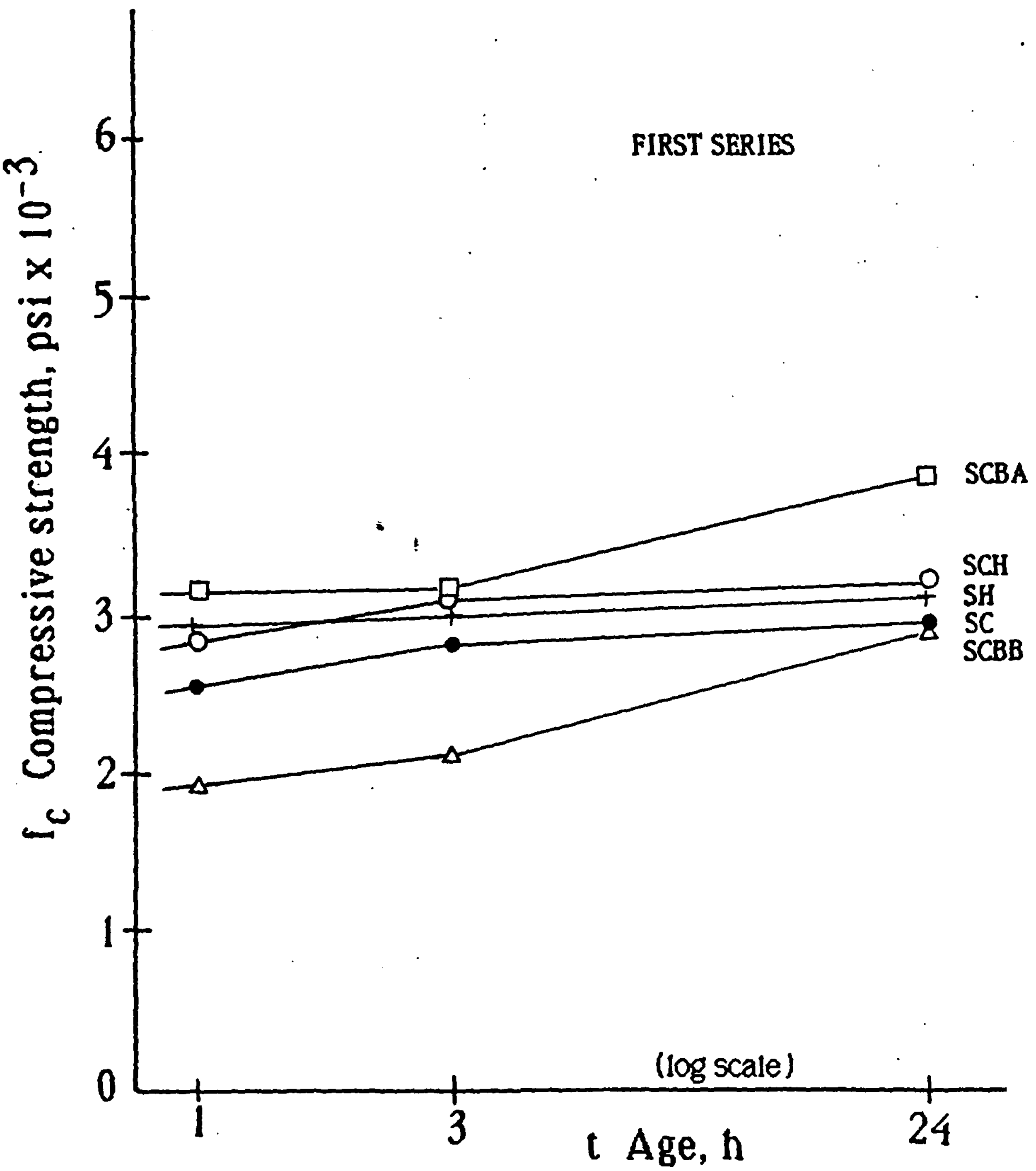


Fig. 4 - Relationship between compressive strength and the early age of SET-45 mortars air-cured at ambient temperature

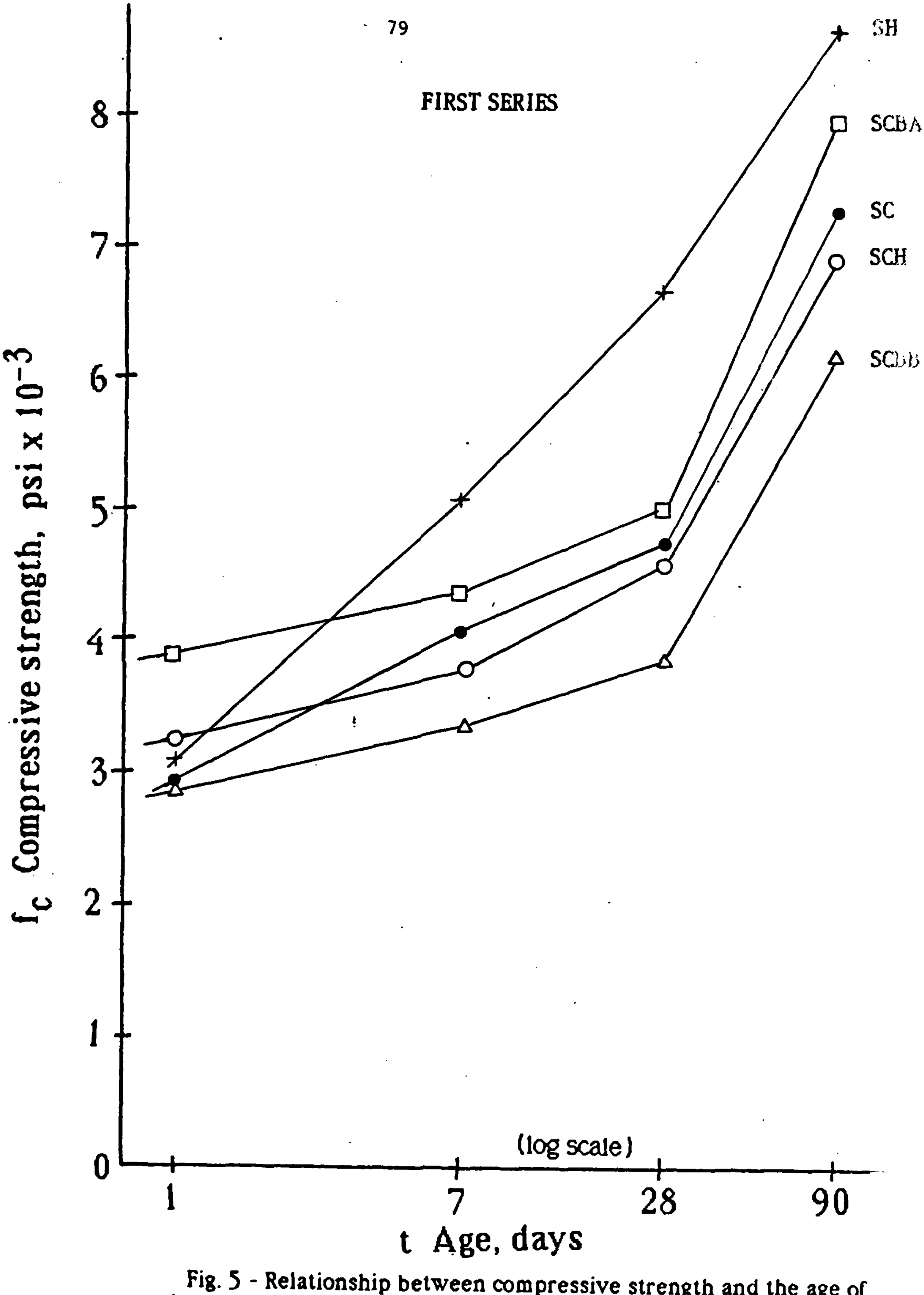


Fig. 5 - Relationship between compressive strength and the age of SET-45 mortars air-cured at ambient temperature

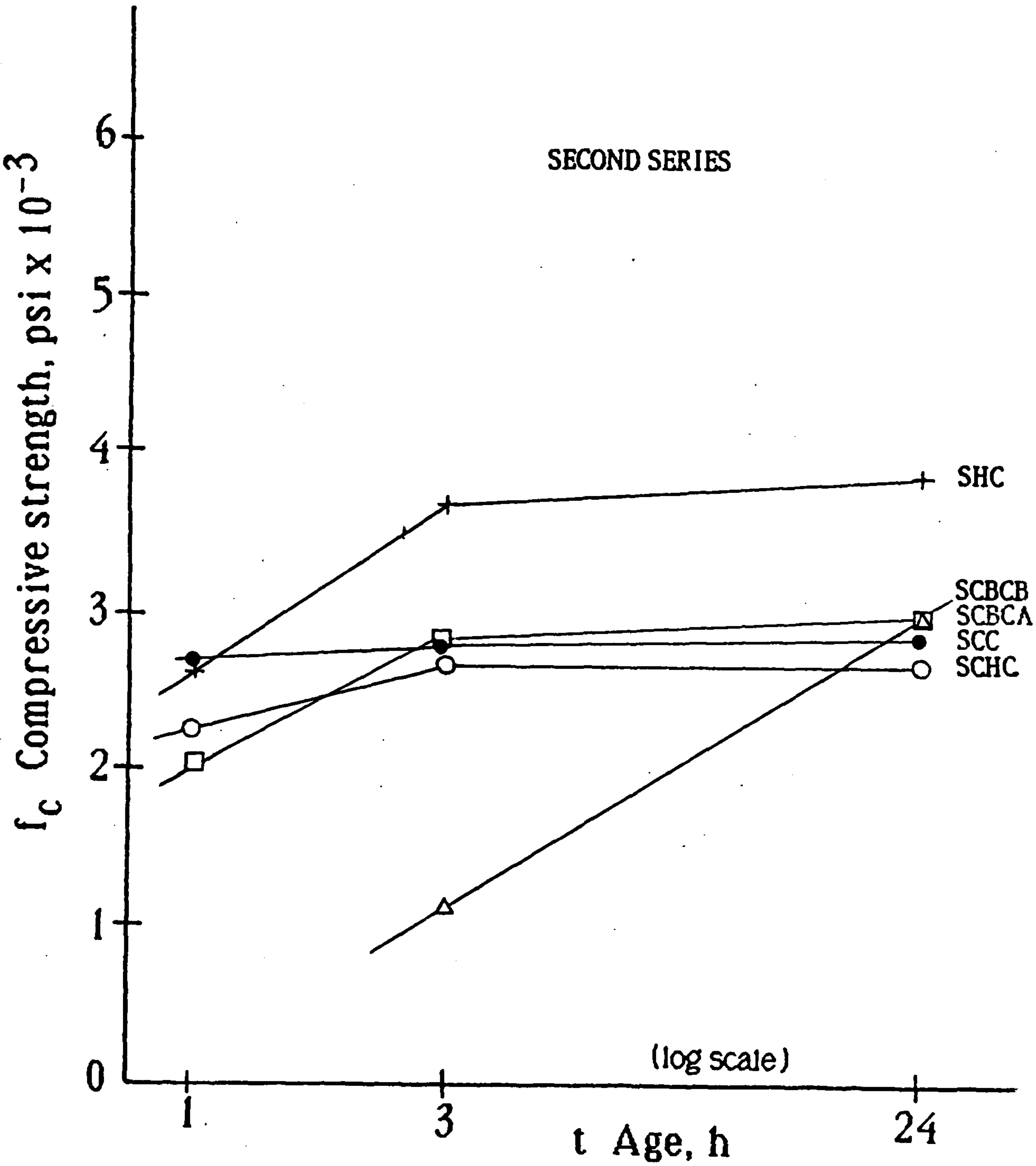


Fig. 6 - Relationship between compressive strength and the early age SET-45 mortars air cured at 32°F (0°C)

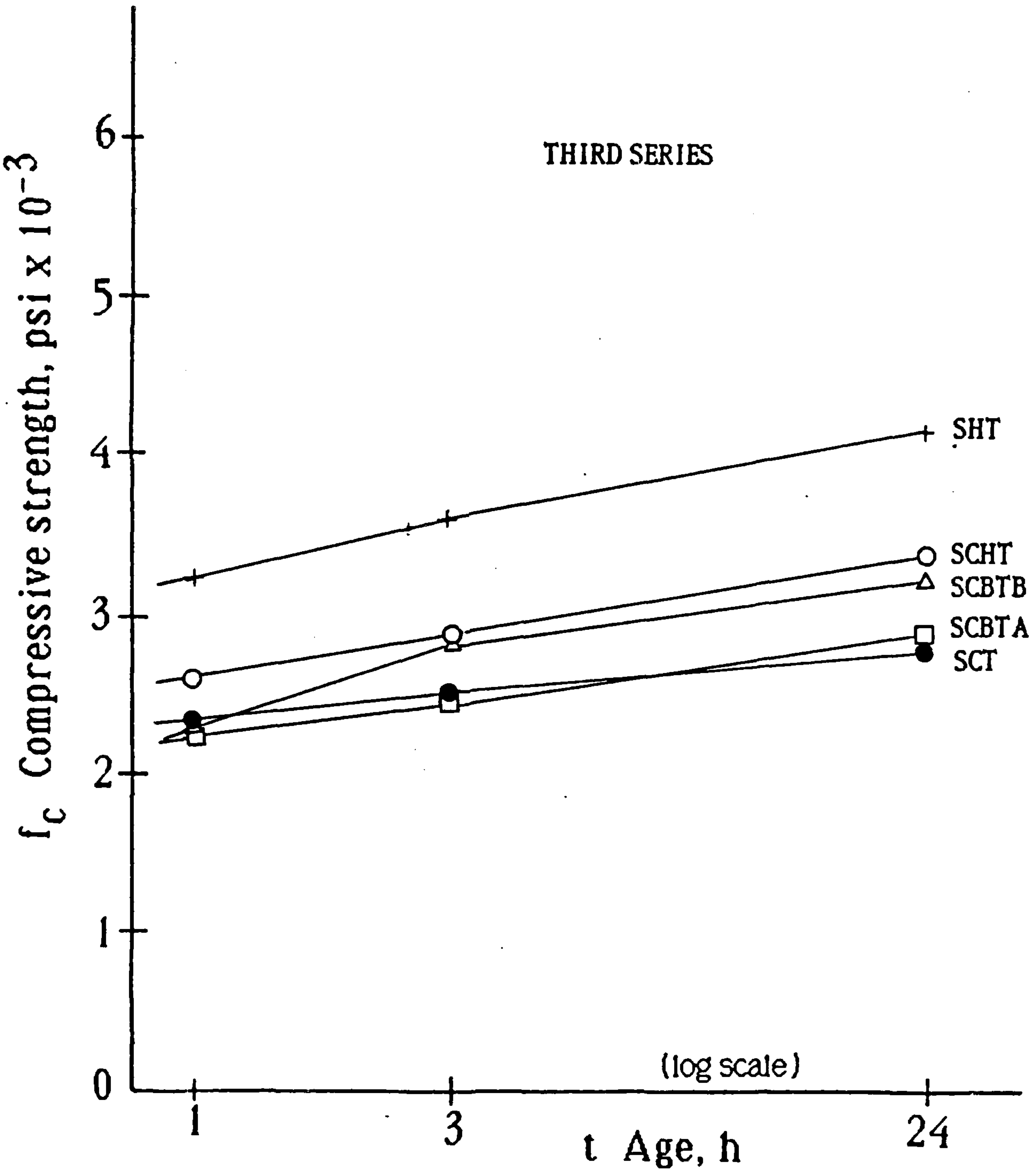


Fig. 7 - Relationship between compressive strength and the early age of SET-45 mortars air cured at 100°F (39°C)

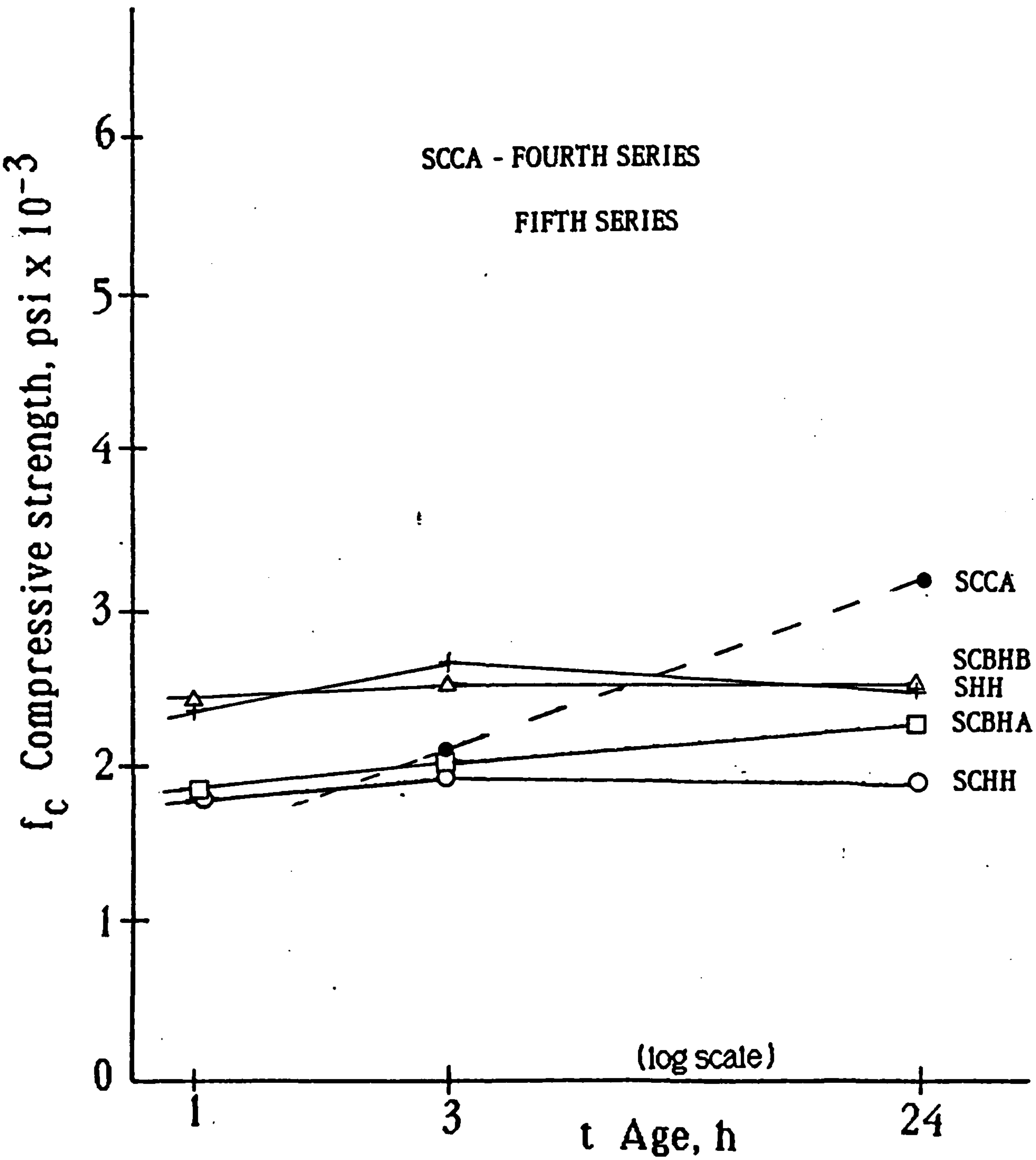


Fig. 8 - Relationship between compressive strength and the early age of SF[illegible]-45 mortars made with preheated materials and air cured at 100°F (3[illegible]

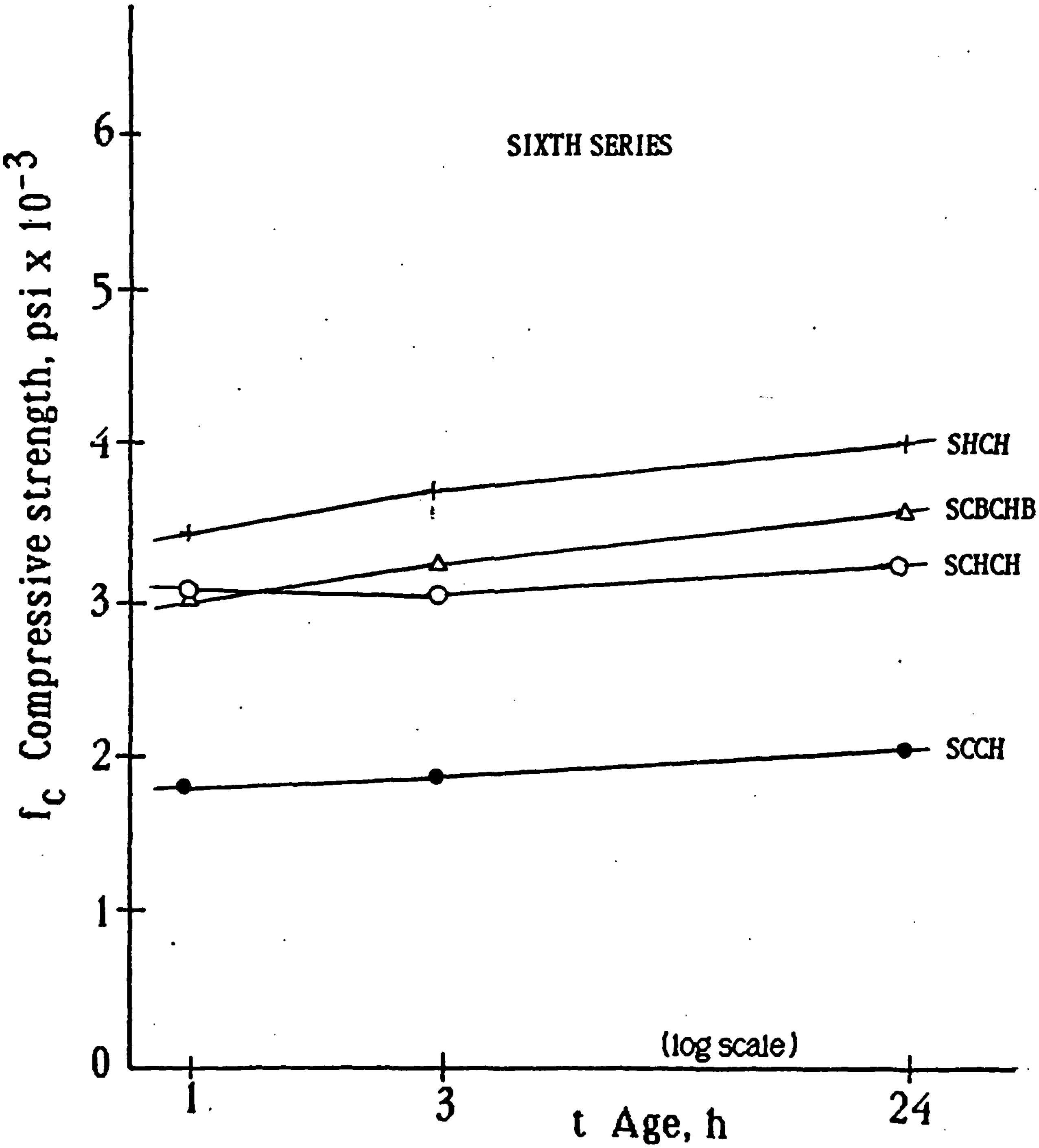


Fig. 9 - Relationship between compressive strength and the early age of SET-45 mortars made with preheated water and air cured at 0°

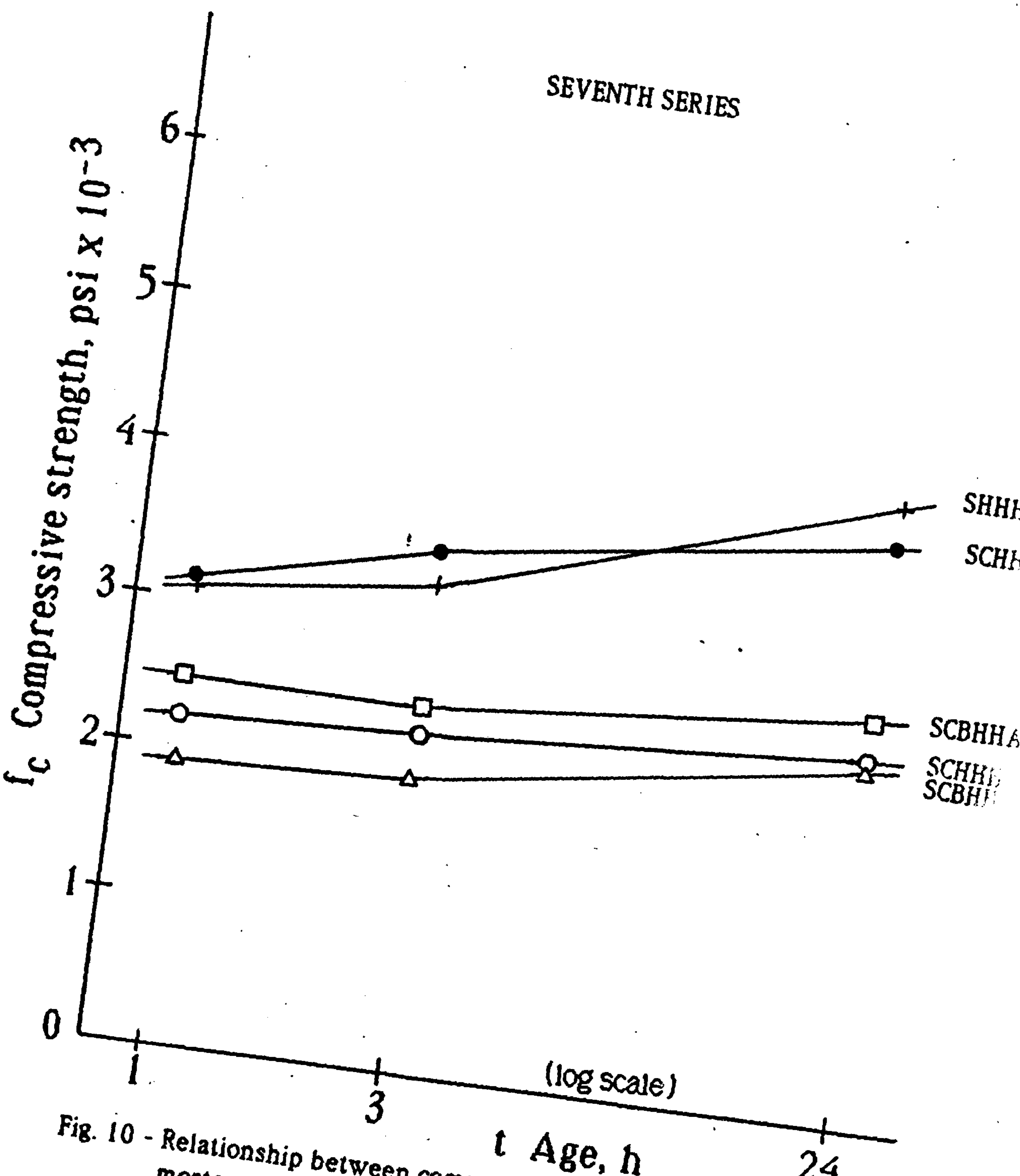


Fig. 10 - Relationship between compressive strength and the early age of ... mortars made with preheated water and air cured at 100°F (39°C

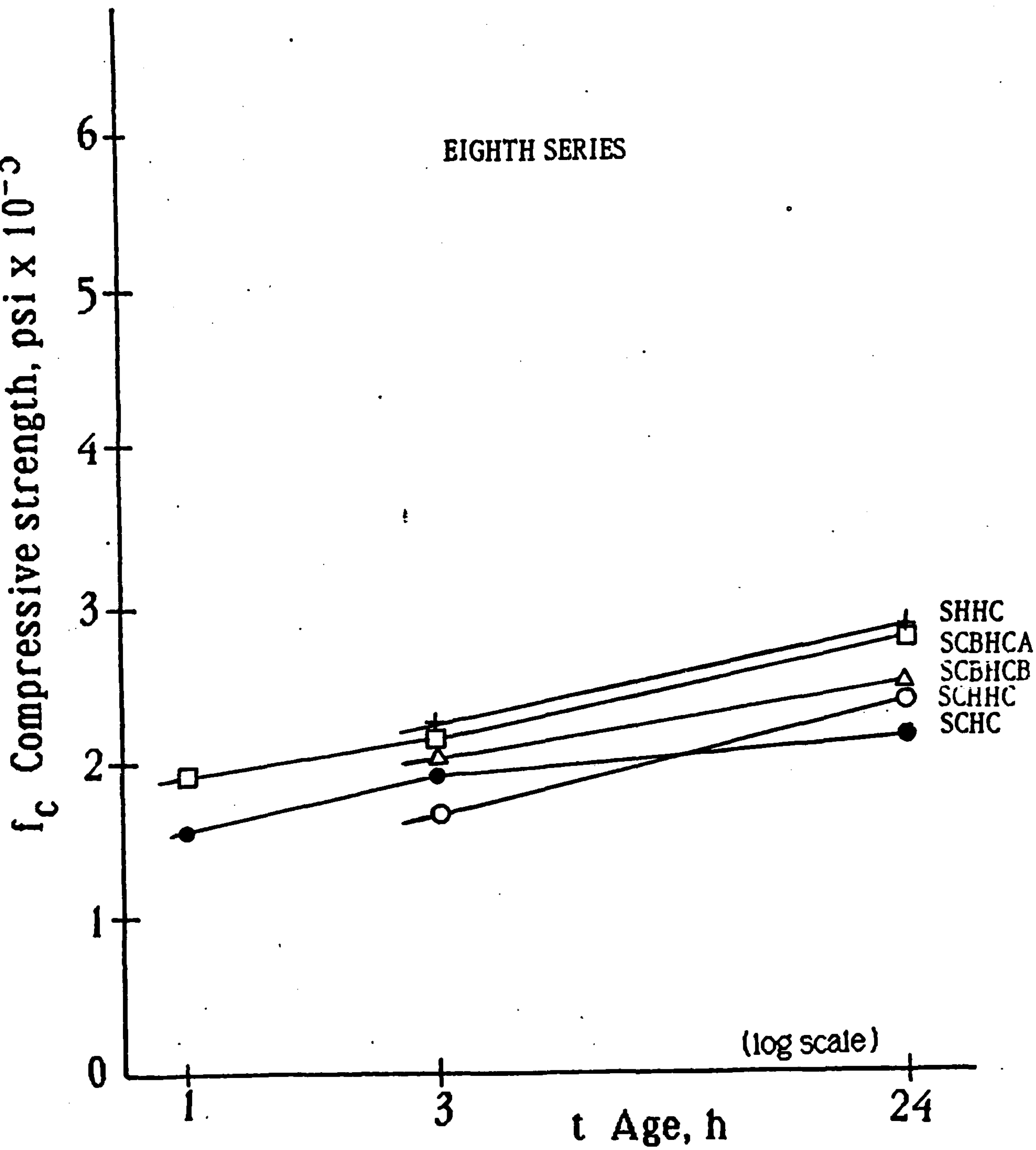


Fig. 11 - Relationship between compressive strength and the early age of SE 45 mortars made with precooled water and air cured at 100°F (39°C)

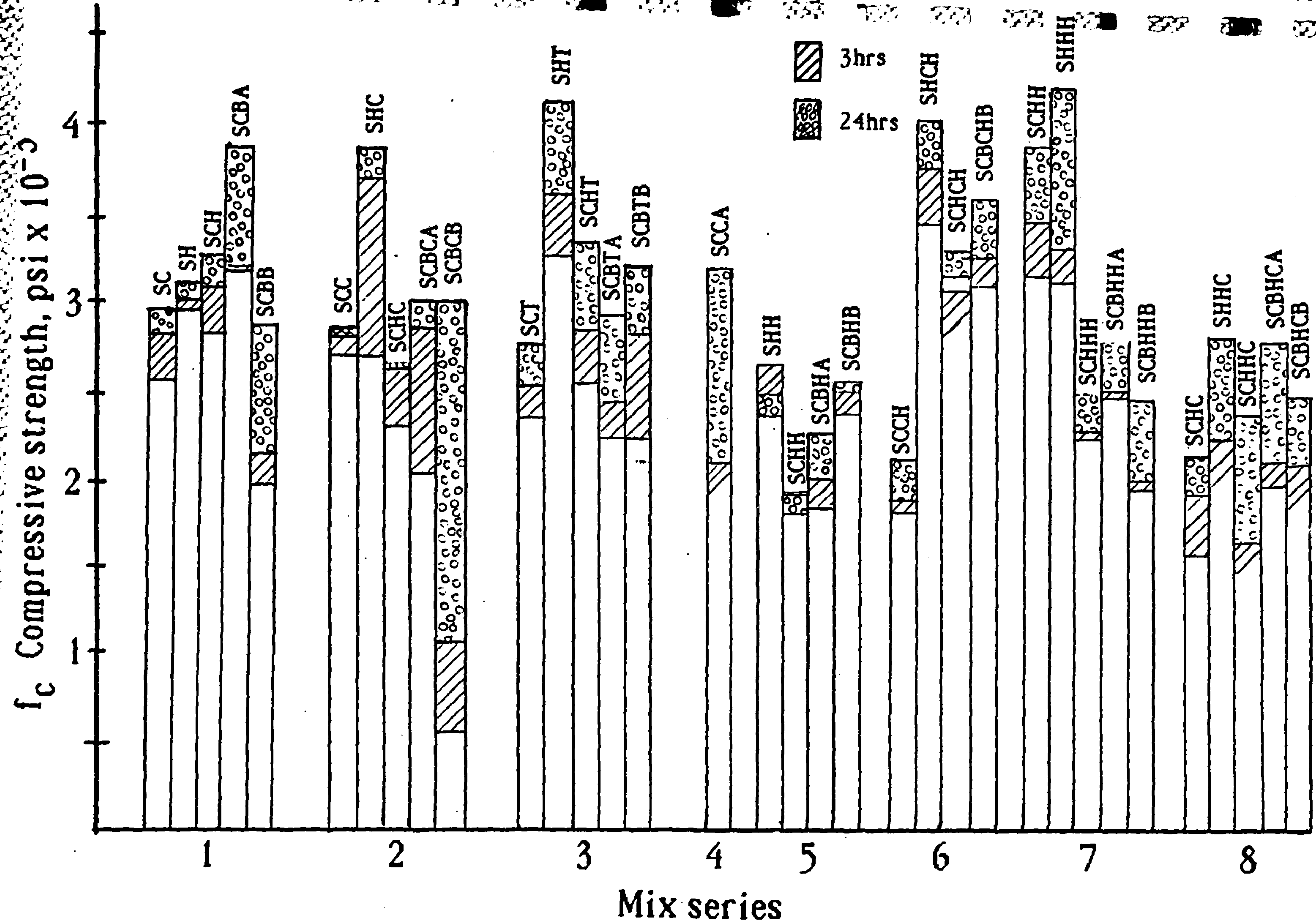


Fig. 12 - Early age compressive strength of SET-45 mortars at various dry mixtures, mixing water, and curing temperatures

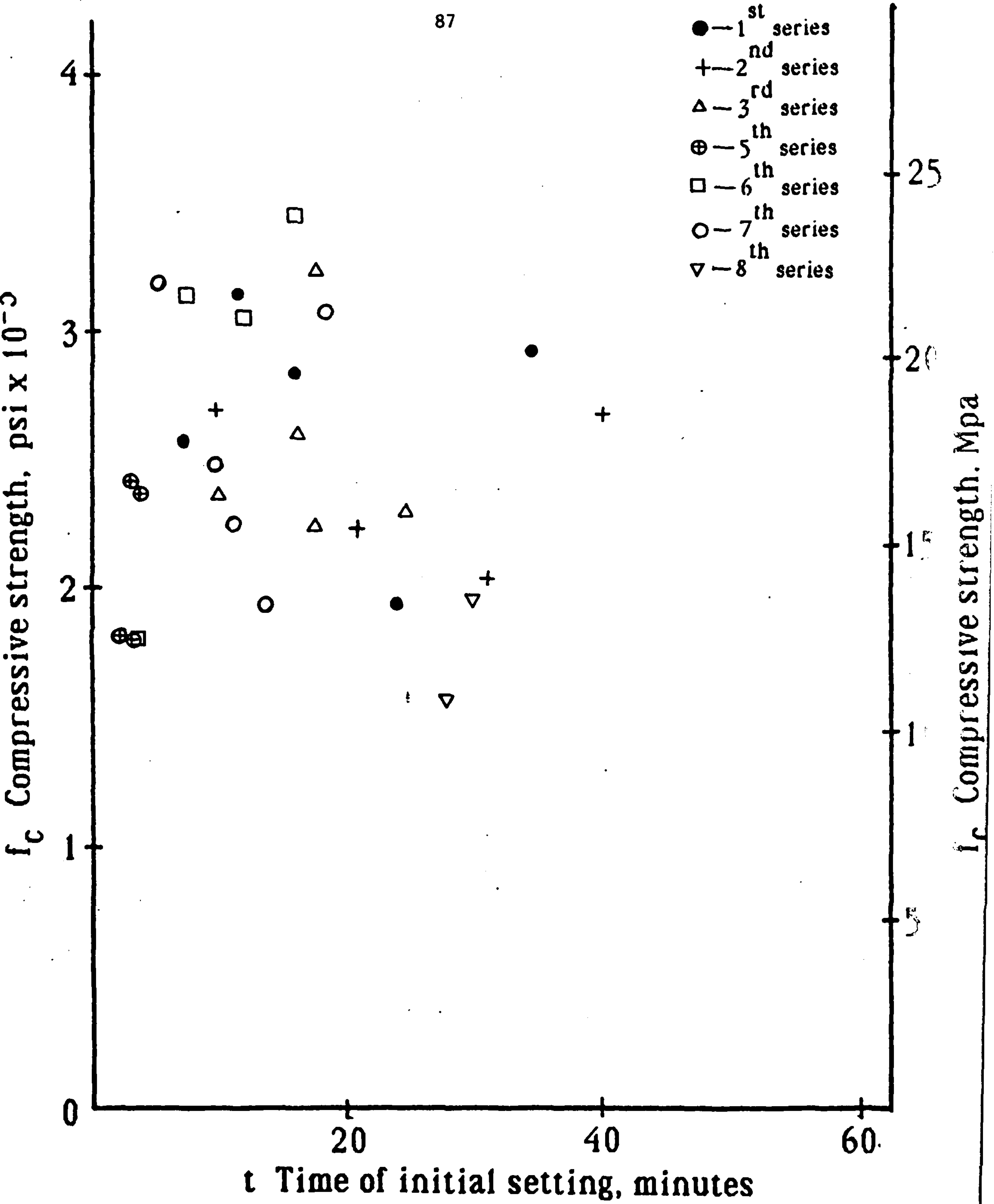


Fig. 13 - Relationship between one hour compressive strength and time of initial setting of SET-45mortars at various dry mixture,mixing water and curing temperatures

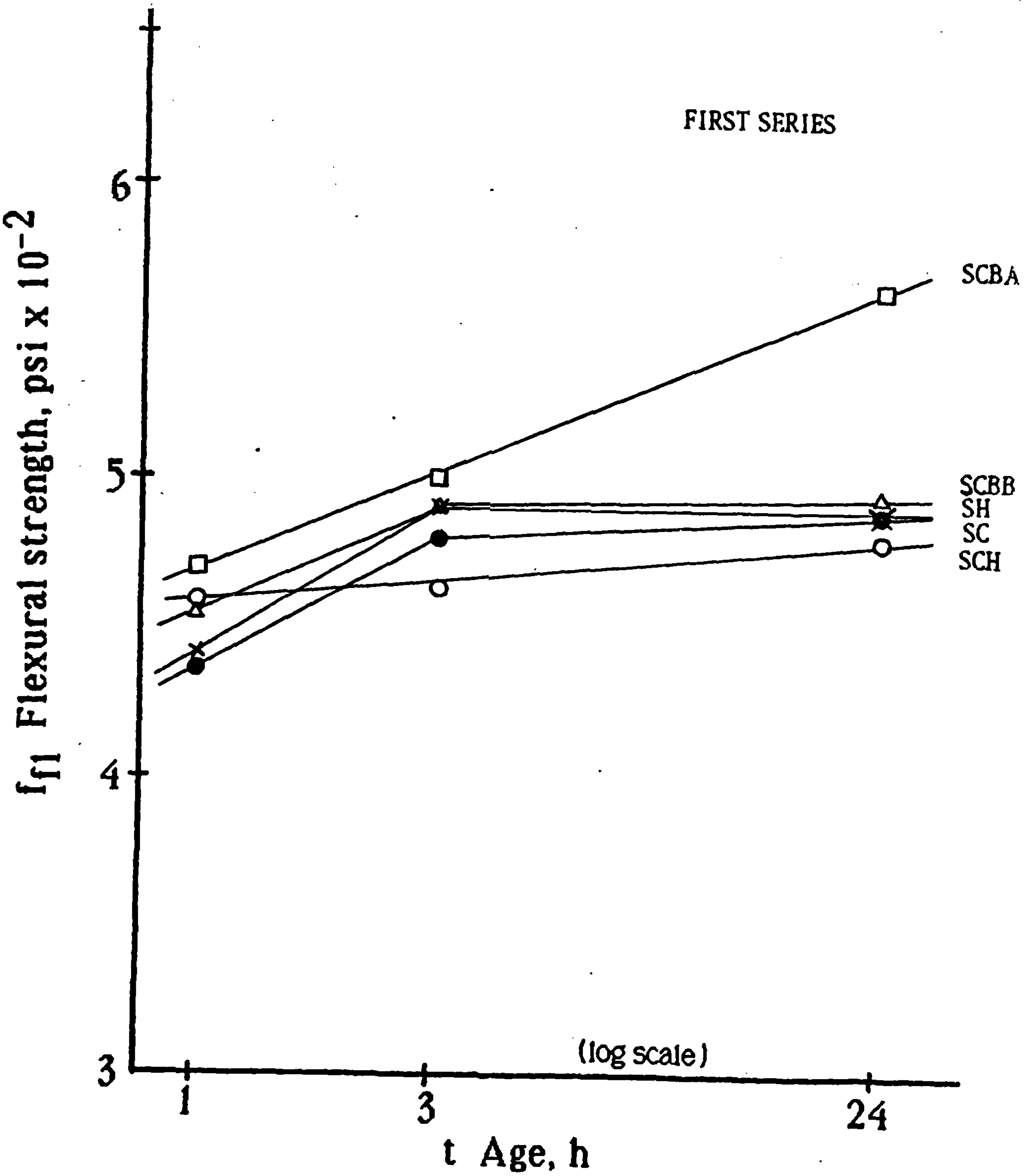


Fig. 14 - Relationship between flexural strength and the early age of SET-45 mortars air cured at ambient temperature

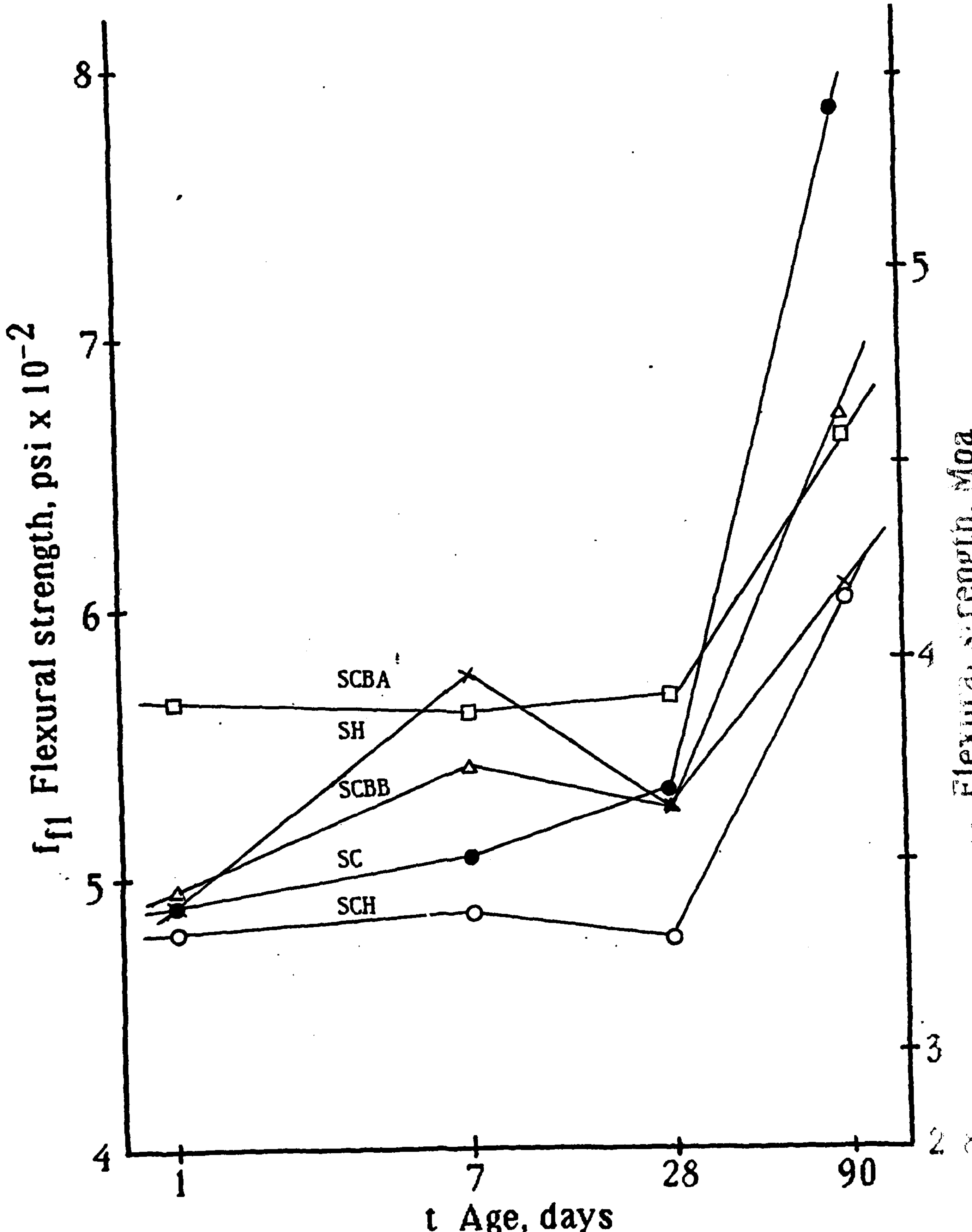


Fig. 15 - Relationship between flexural strength and the age of SET-45 mortars air cured at ambient temperature

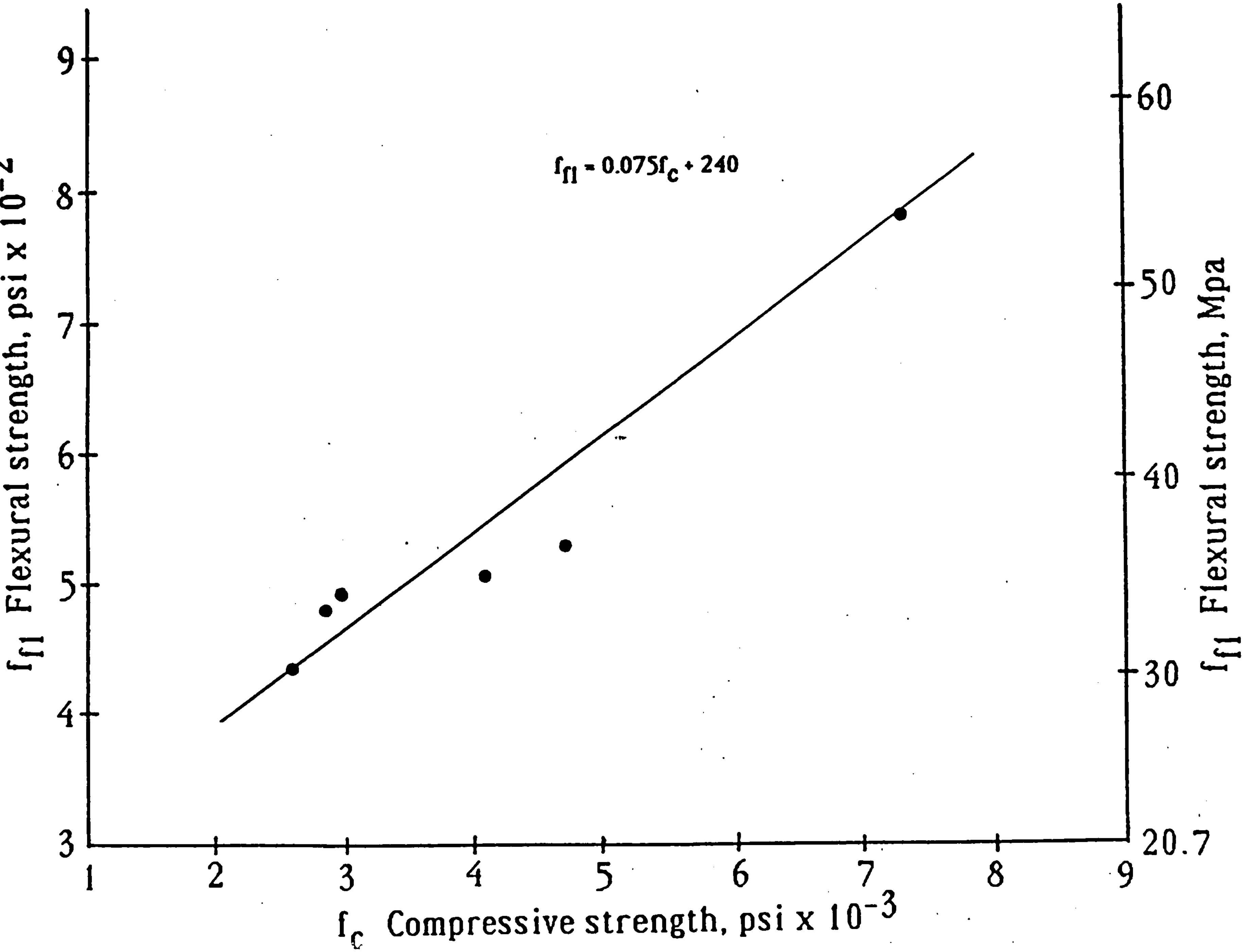


Fig. 16 Relationship between flexural strength and compressive strength of SET-45 cold mortar air cured at ambient temperature

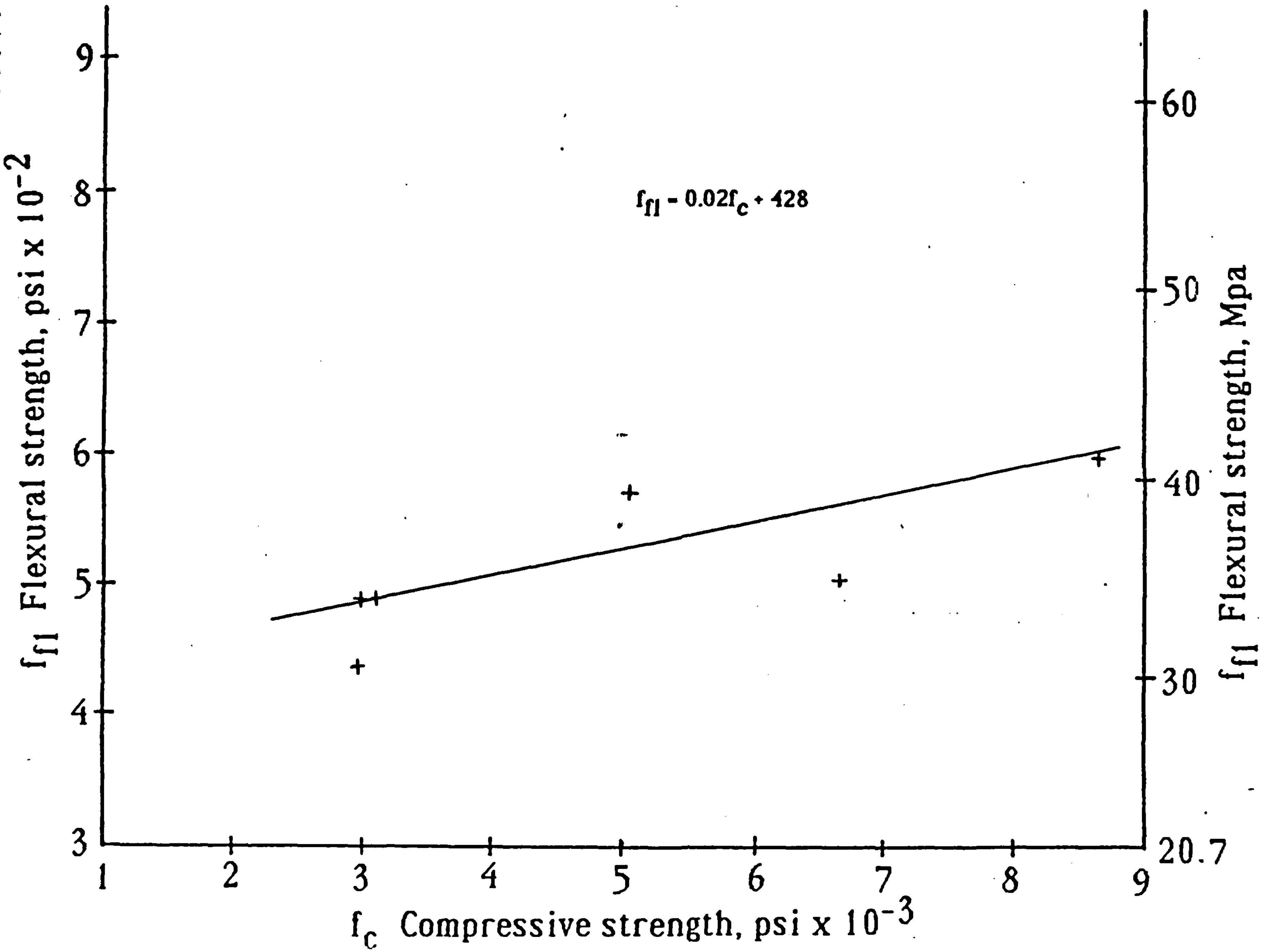
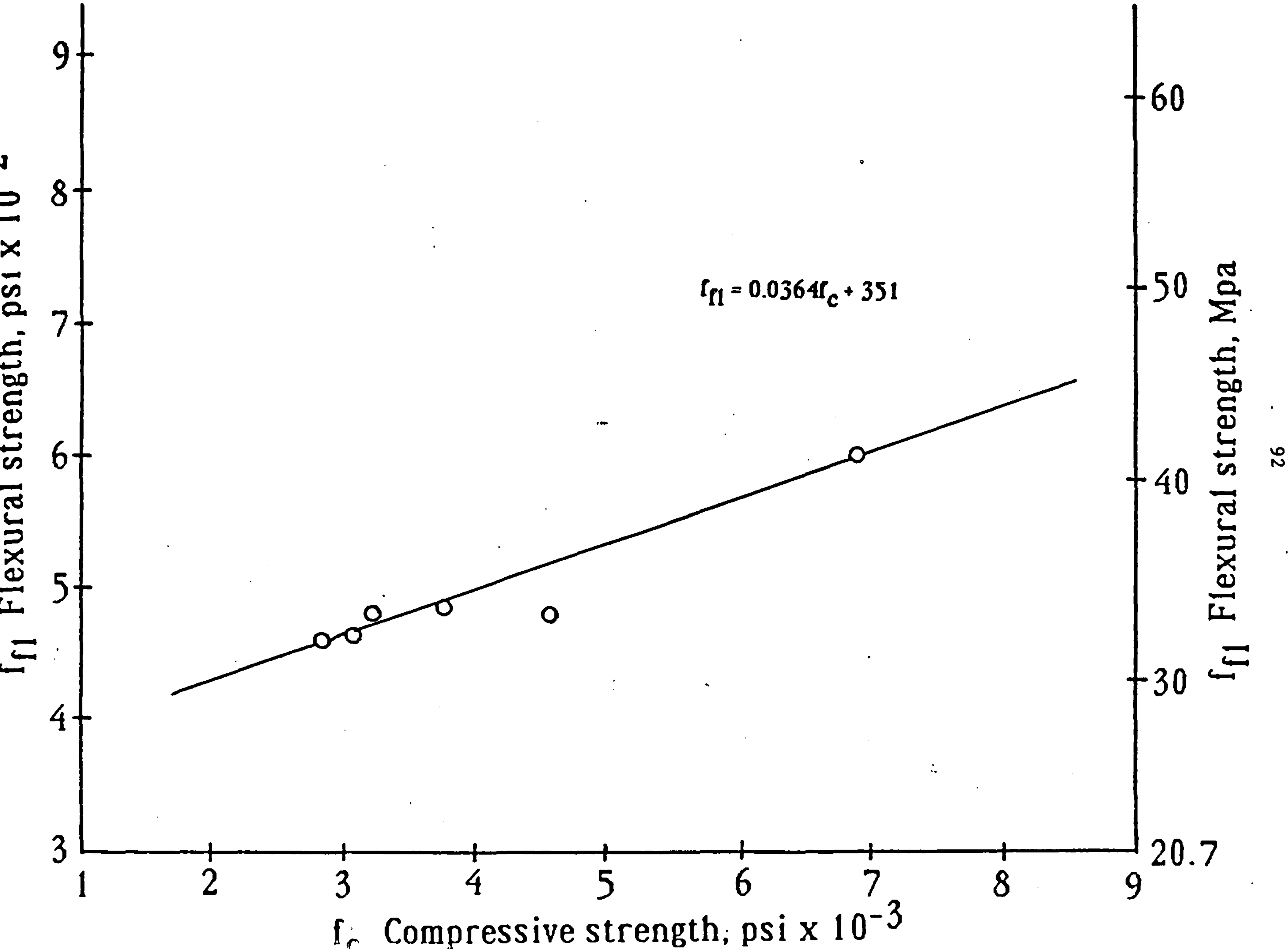


Fig. 17 - Relationship between flexural strength and compressive strength of SET-45 hot mortar air cured at ambient temperature



[illegible] between [illegible] strength and compressive strength of SET-45 cold + hot mortar air cured at ambient temperature

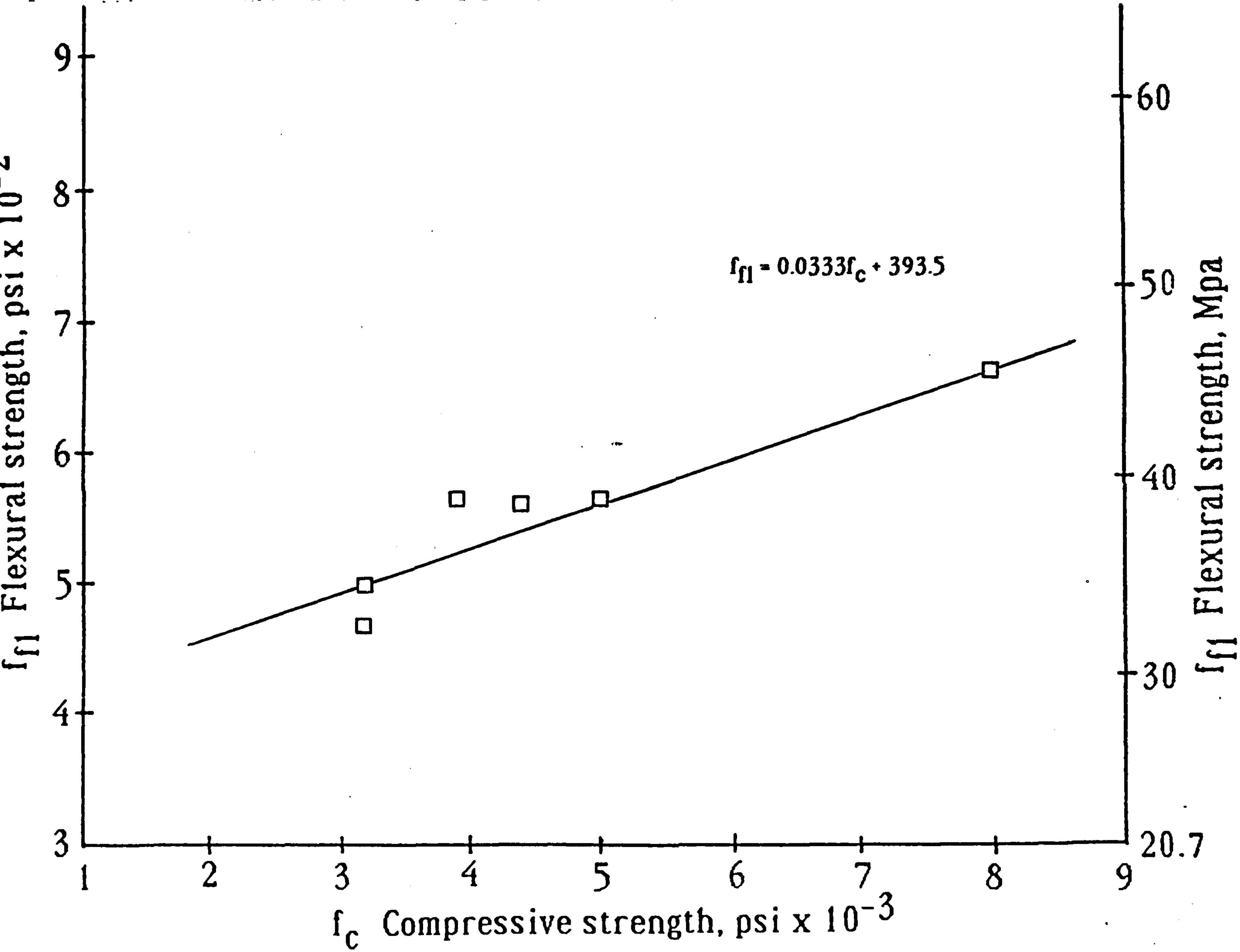


Fig [illegible] Relationship between flexural strength and compressive strength of SET-45 [illegible] borax (0.34%) mortar air cured at ambient temperature

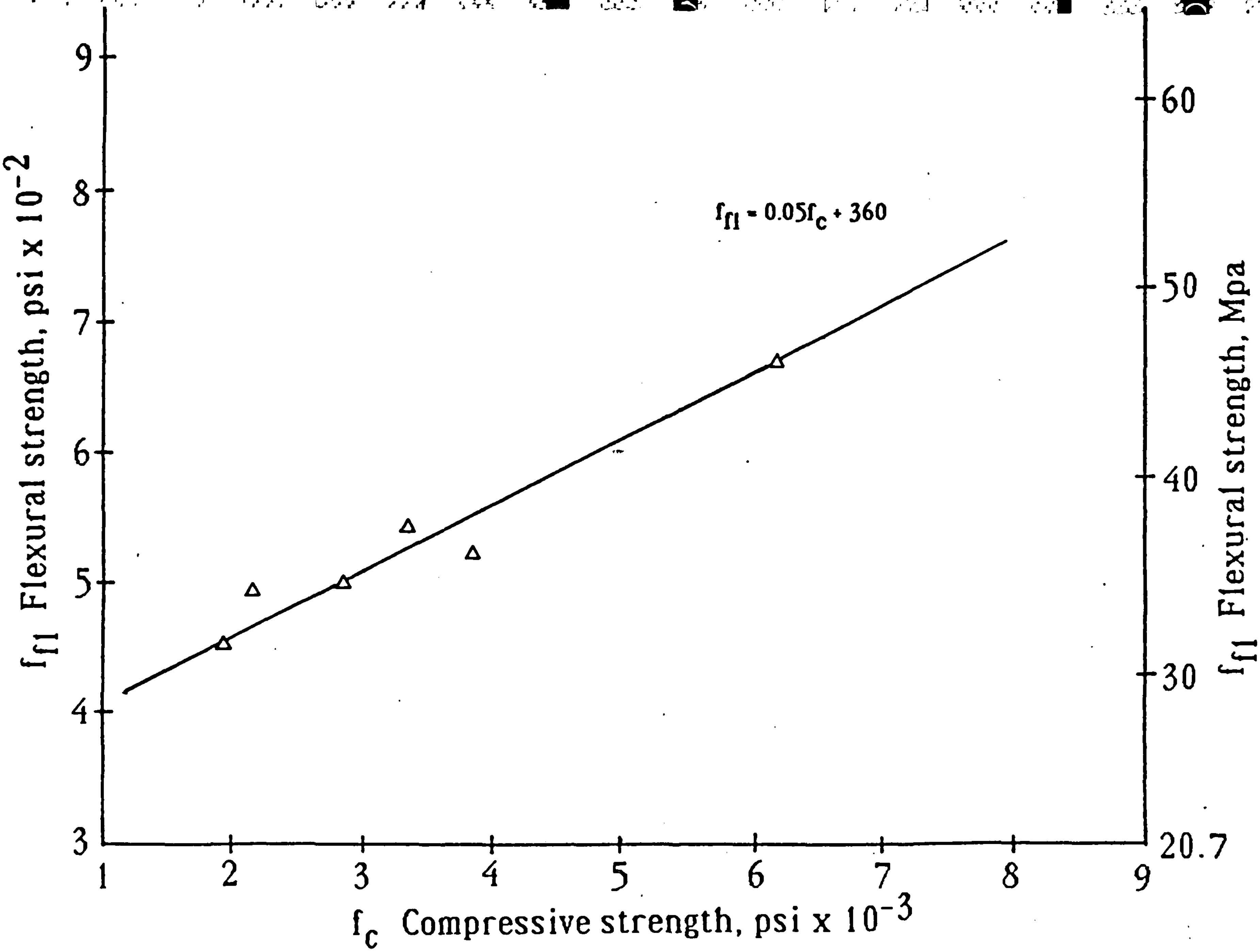


Fig 20 - Relationship between flexural strength and compressive strength of [illegible] borax (0.7[illegible] [illegible] air cured at ambient temperature

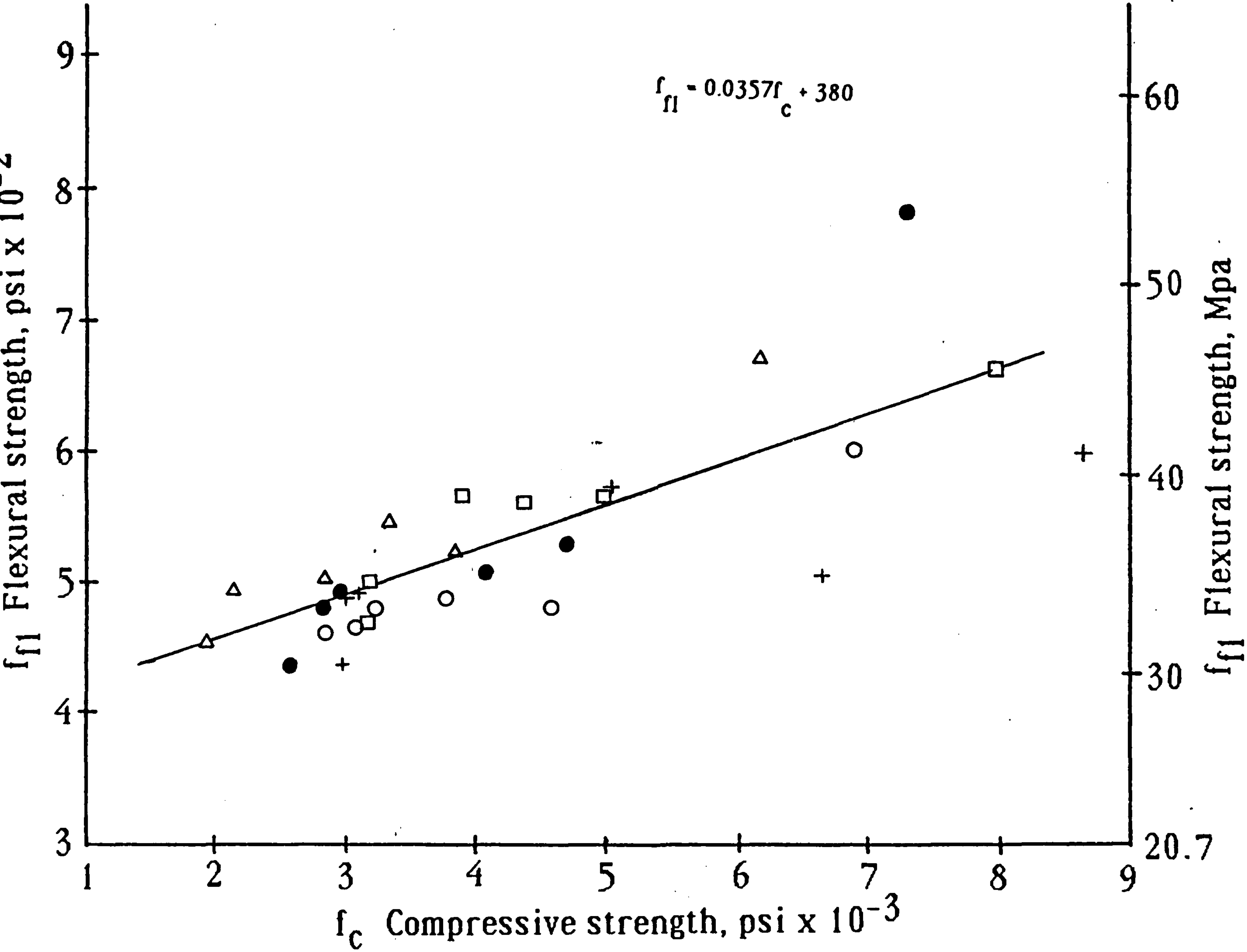


Fig. 21 - Relationship between flexural strength and compressive strength of SET-45 mortars air cured at ambient temperatures. (Summary)

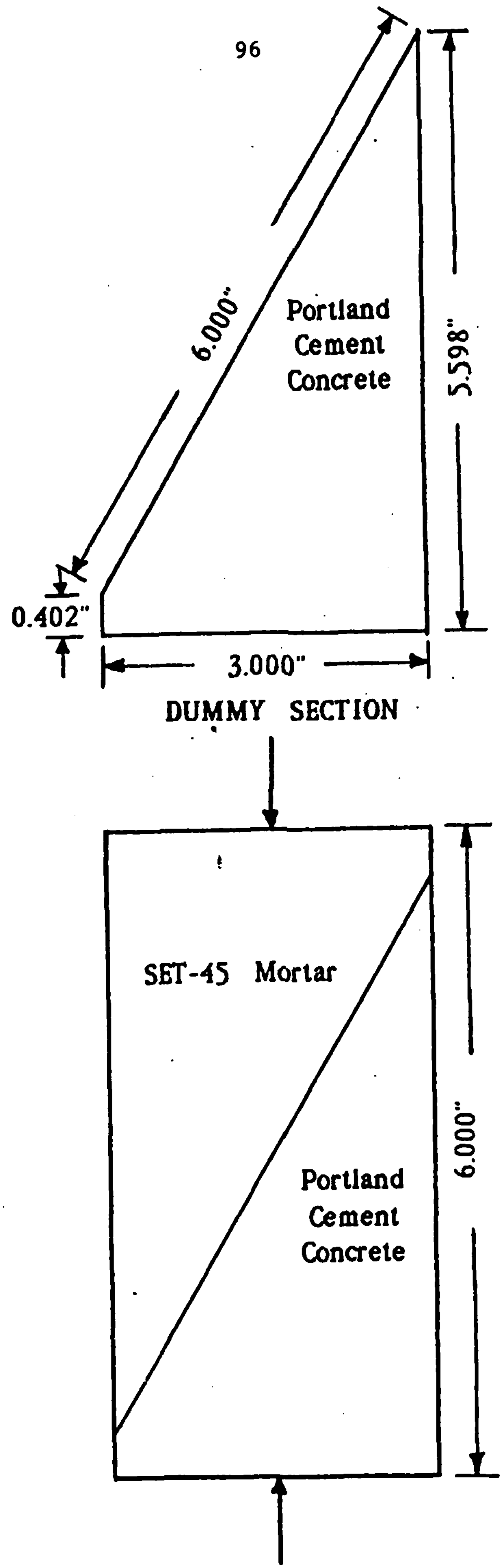


Fig. 22 - Typical shear bond cylindrical specimen

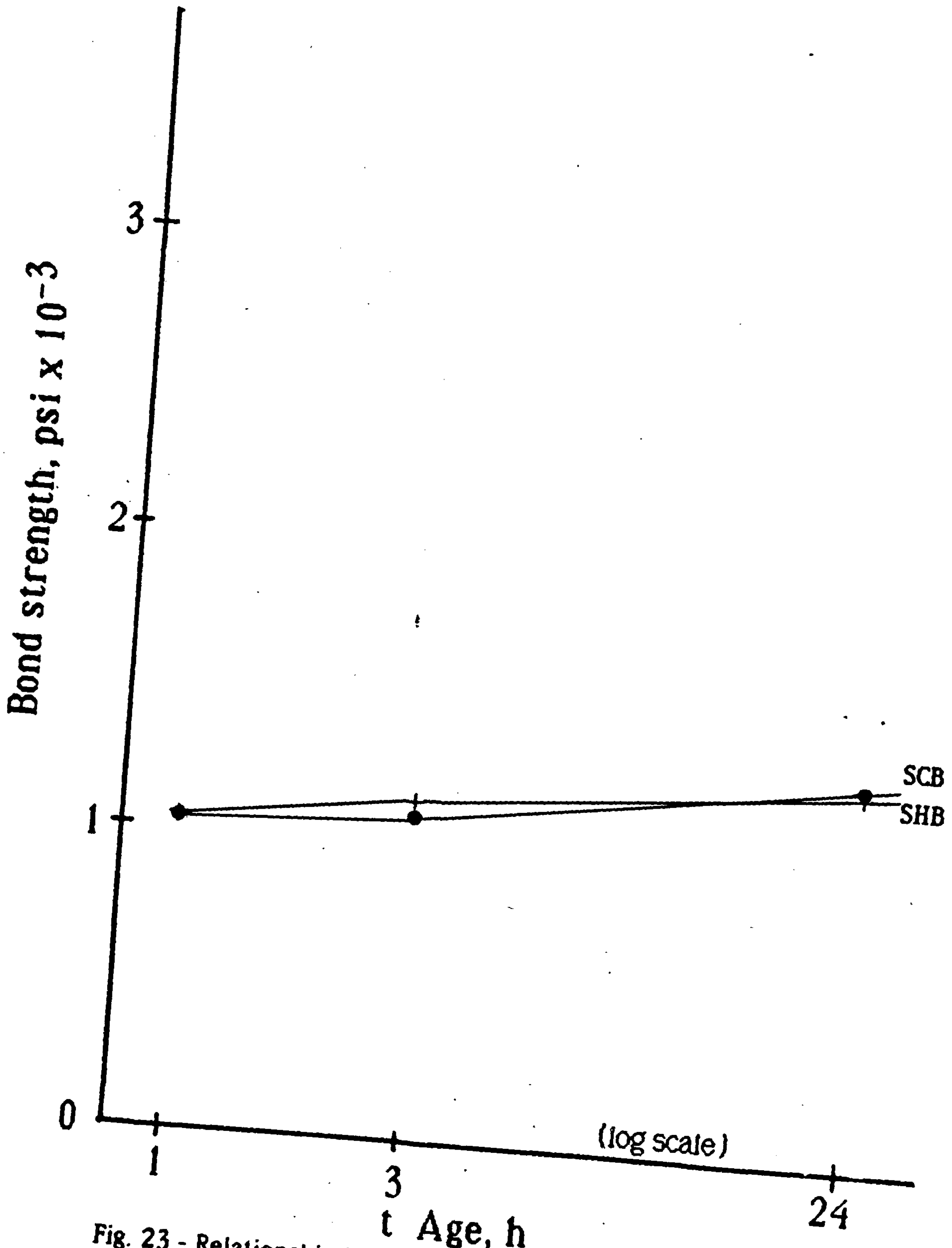


Fig. 23 - Relationship between bond strength and early age of SET-45 cold and hot mortars air cured at ambient temperature

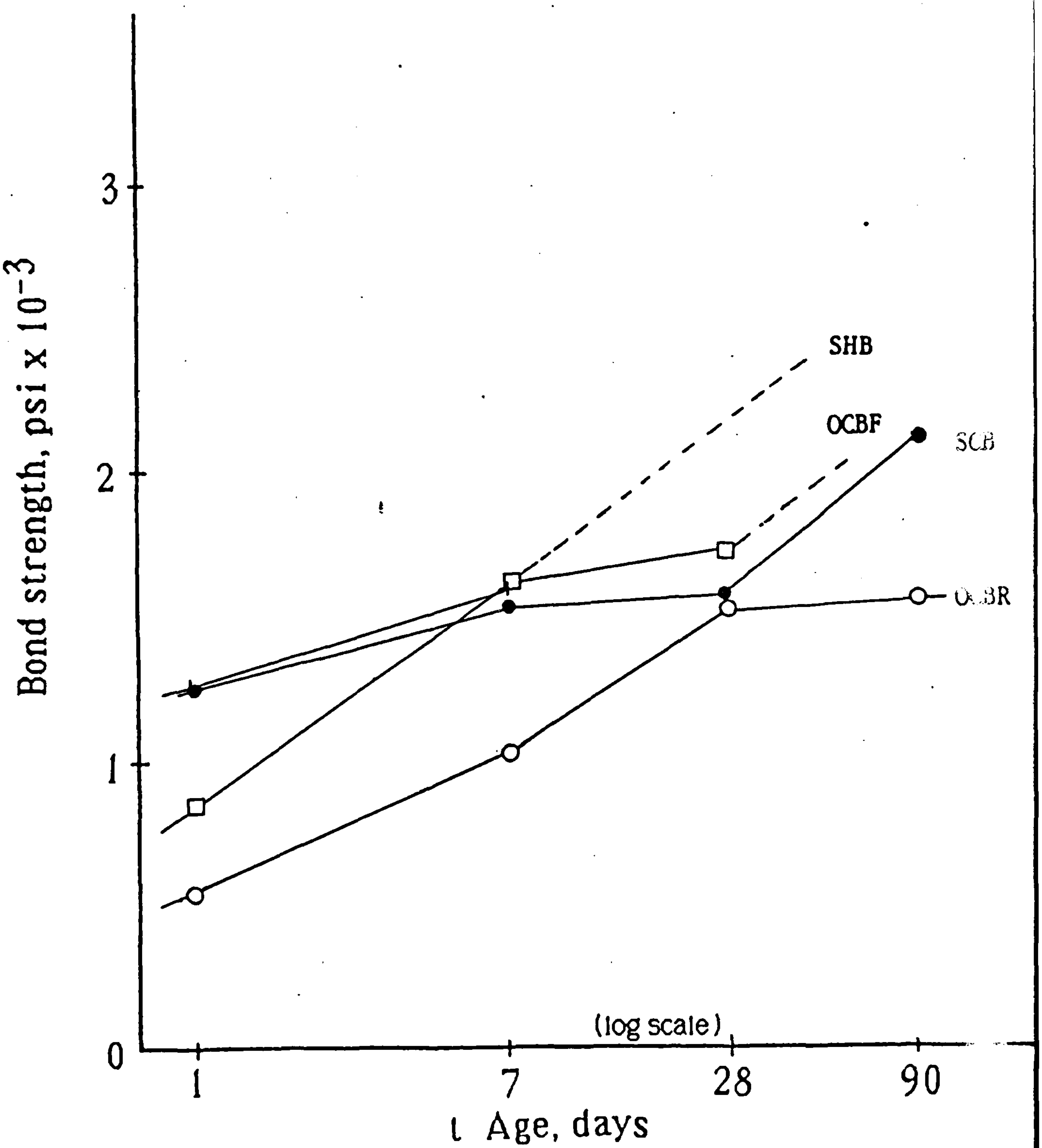


Fig. 24 - Relationship between bond strength and age of SET-45 cold, hot and portland cement concrete. Broken lines

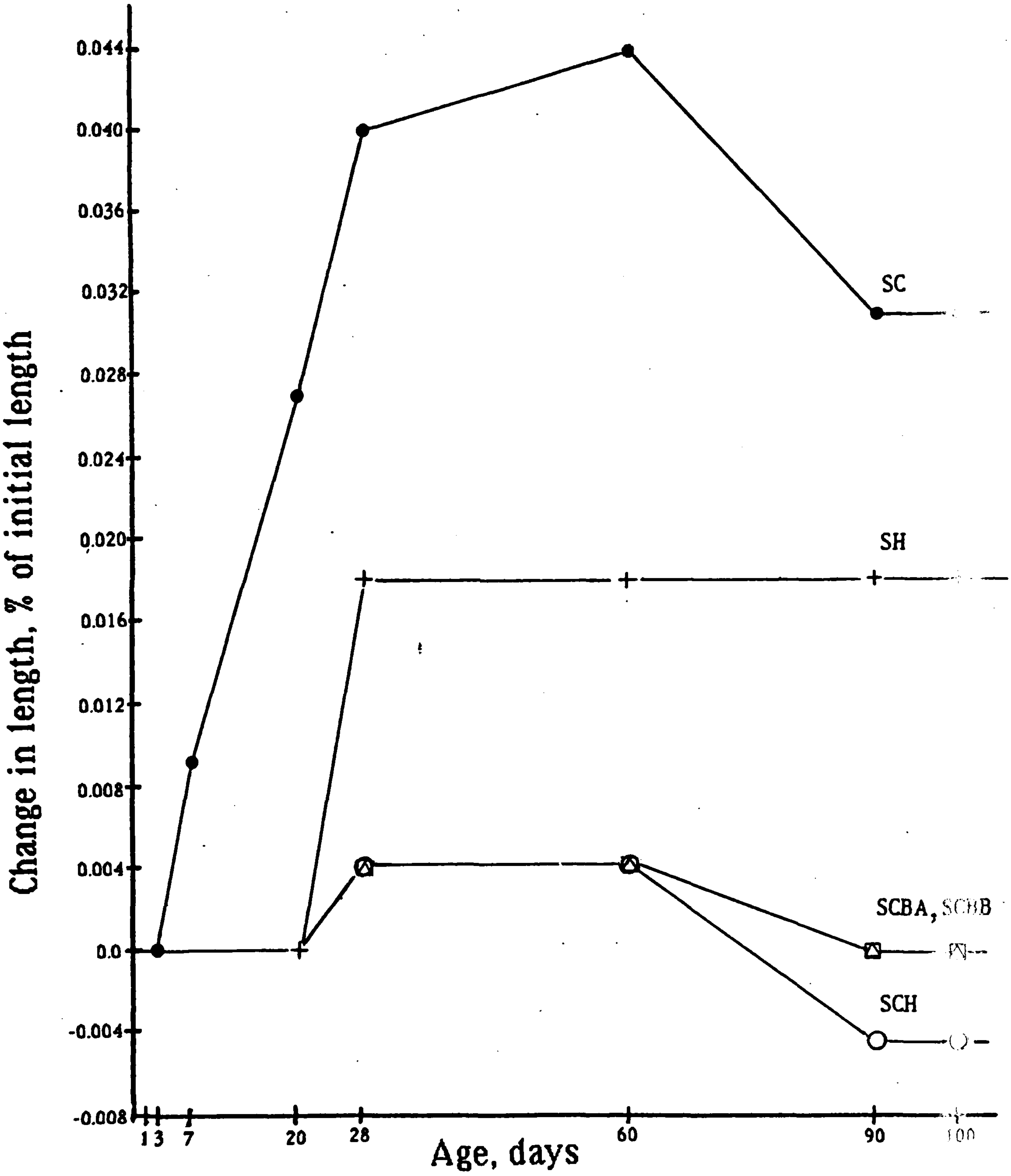


Fig. 25 - Change in length of SET-45 mortars as a function of time (moist cured)

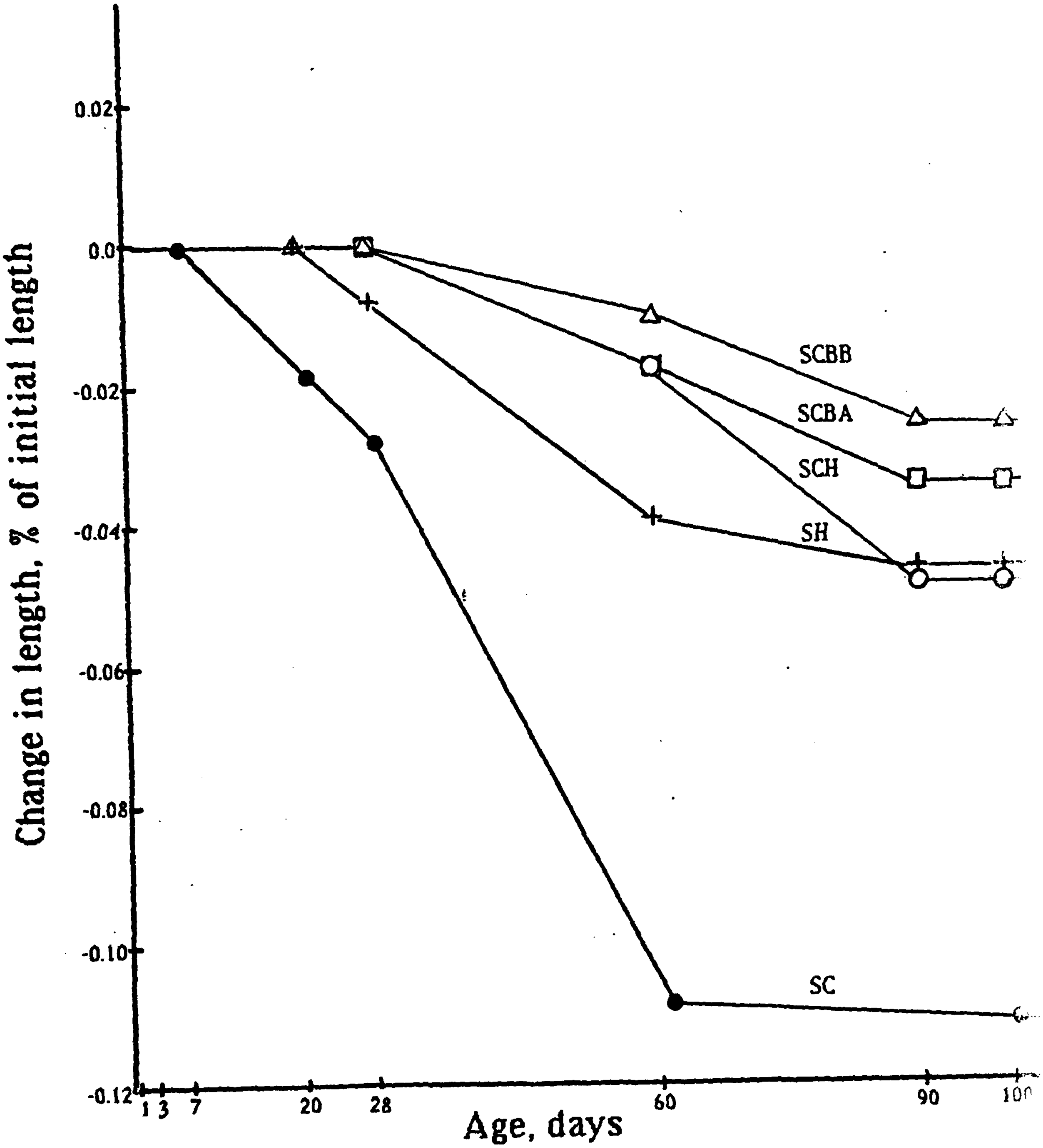


Fig. 26 - Change in length of SET-45 mortars as a function of time (air cured at ambient temperature)

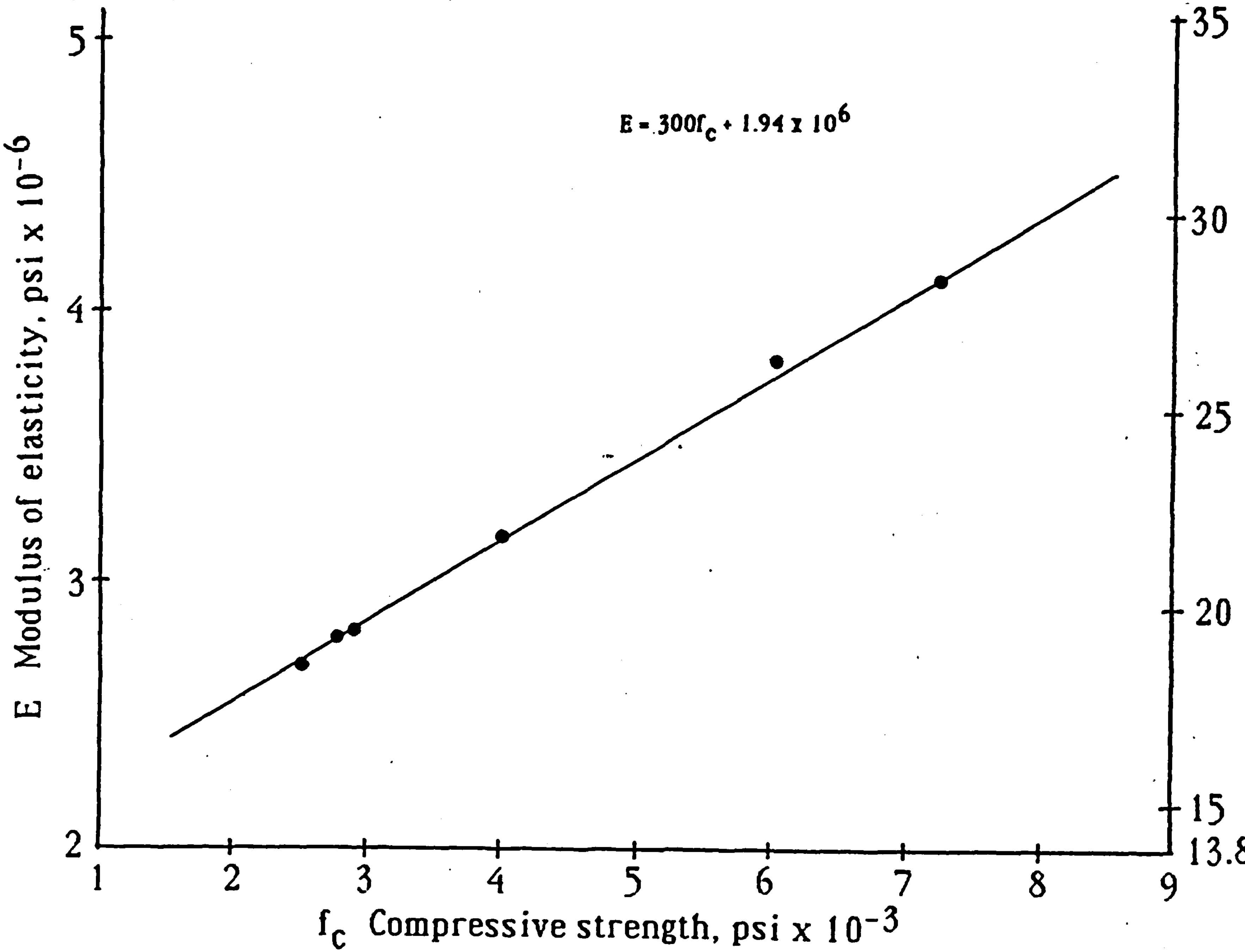


Fig. 27 - Relationship between modulus of elasticity and compressive strength of SPT-45 cold mortar air cured at ambient temperature

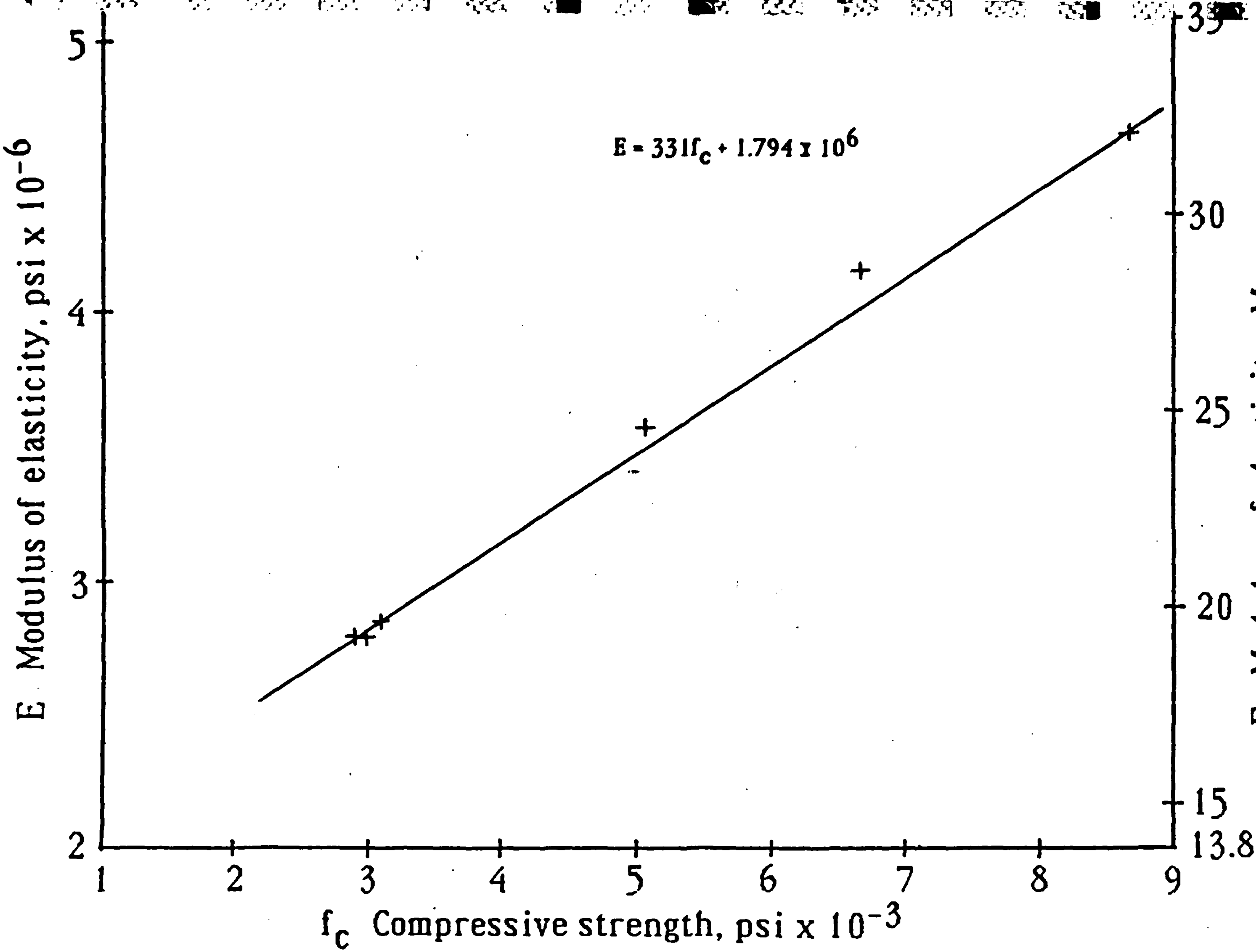


Fig. 28 - Relationship between modulus of elasticity and compressive strength of SET-45 hot mortar air cured at ambient temperature

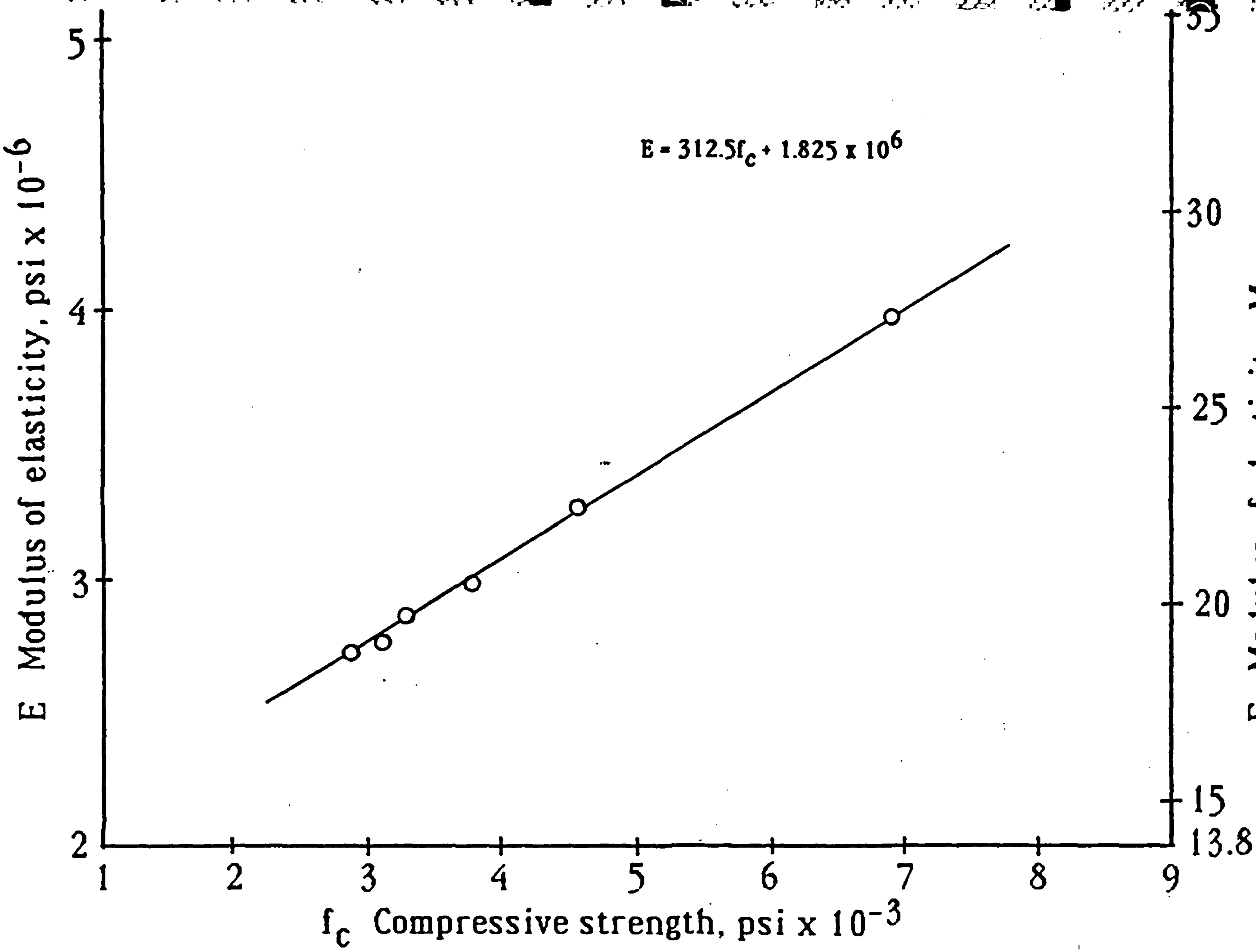


Fig. 29 - Relationship between modulus of elasticity and compressive strength of SET-45 cold + hot mortar air cured at ambient temperature

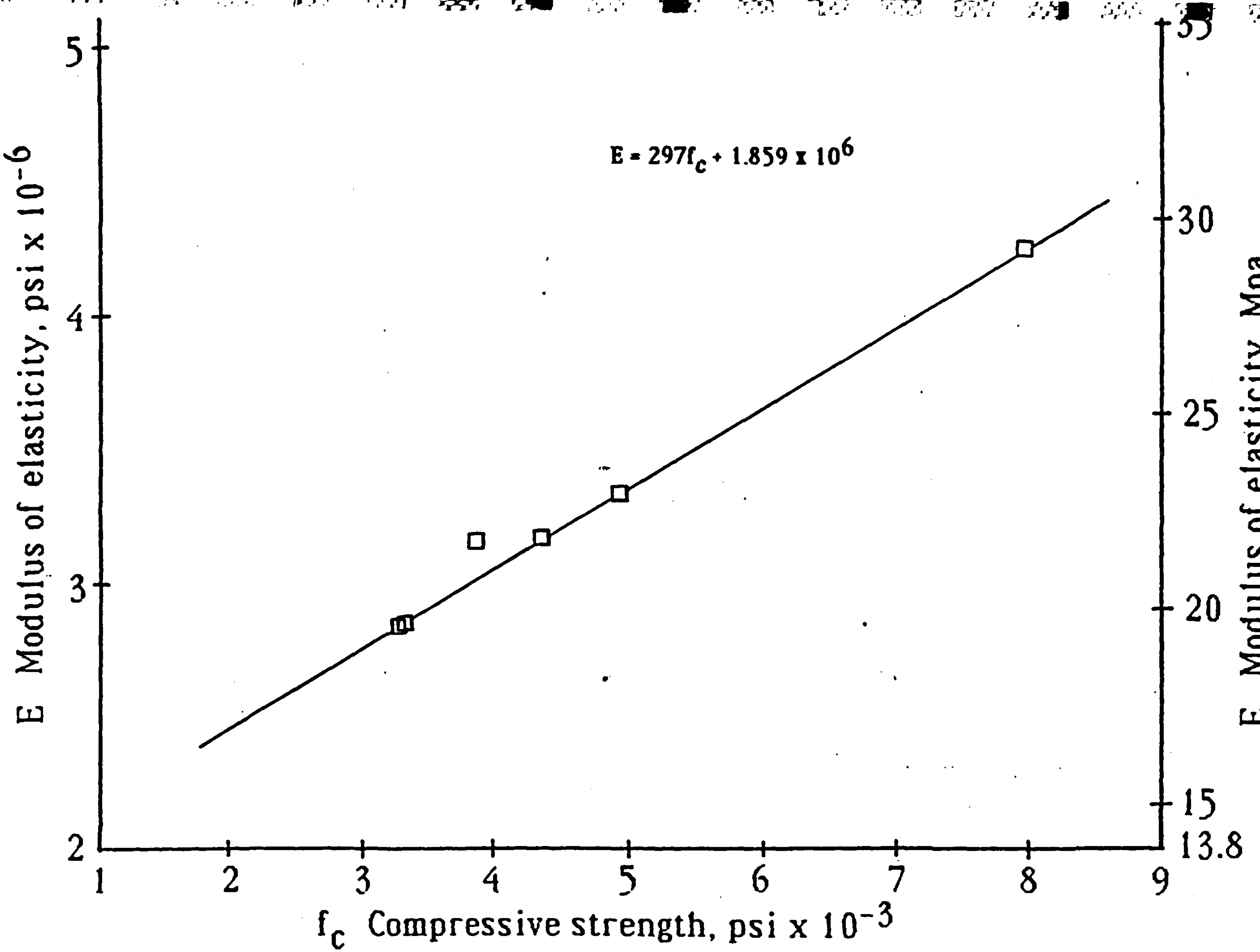


Fig. 30 - Relationship between modulus of elasticity and compressive strength of SET-45 cold + borax (0.34%) mortar air cured at ambient temperature

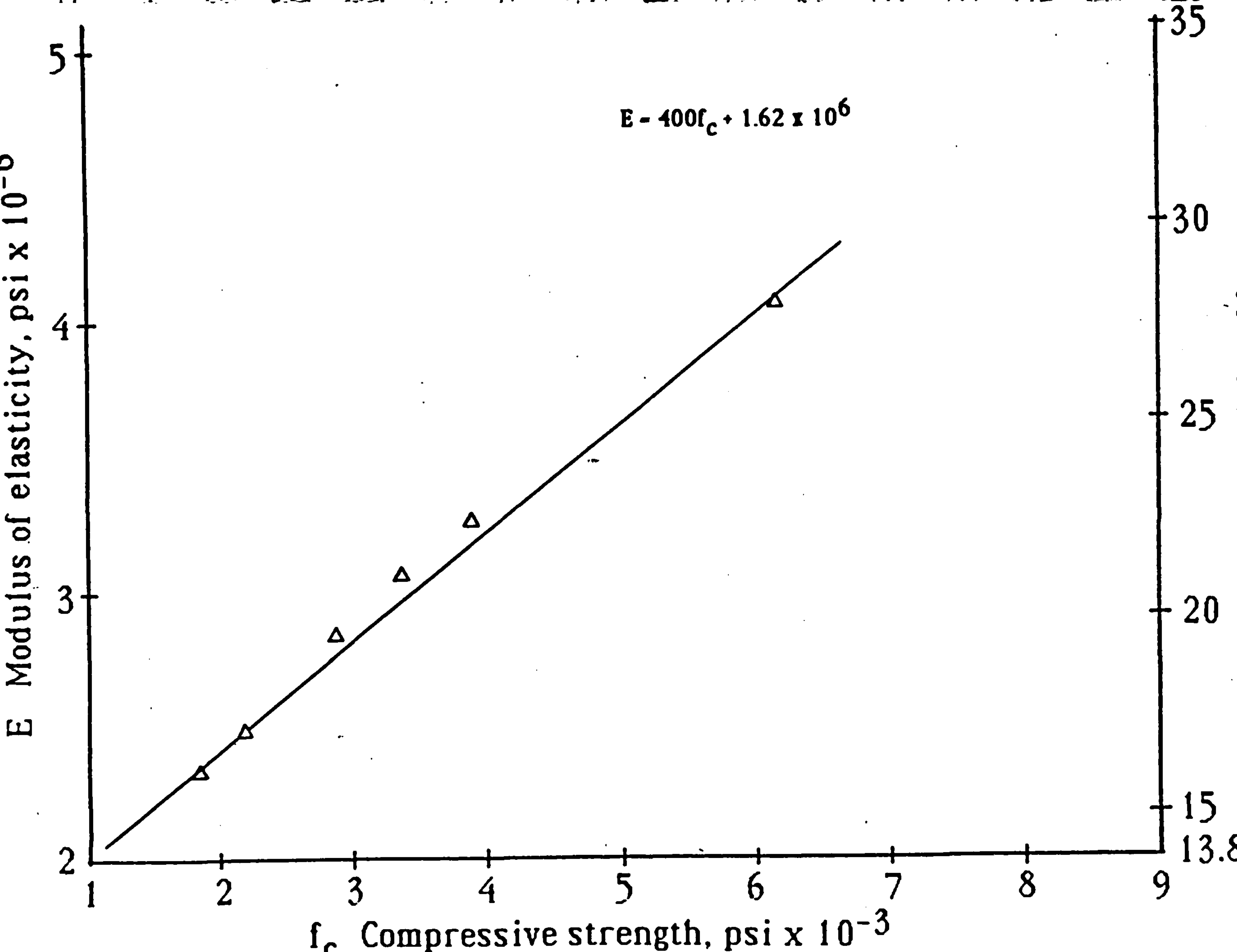


Fig. 31 - Relationship between modulus of elasticity and compressive strength of [illegible] + borax (0.7%) mortar air cured at ambient temperature

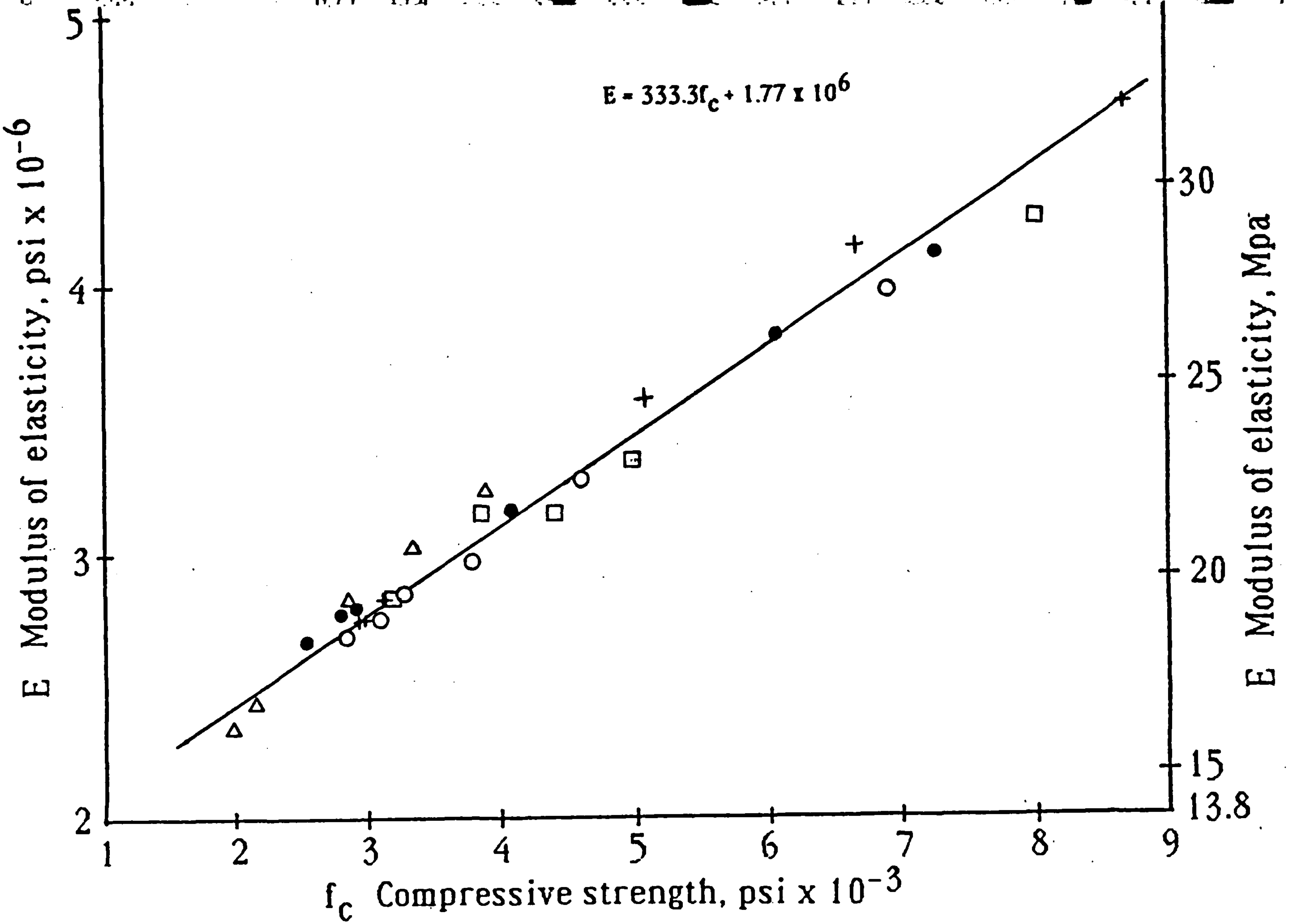


Fig. 32 - Relationship between modulus of elasticity and compressive strength of SET-45 mortars air cured at ambient temperature. (Summary)

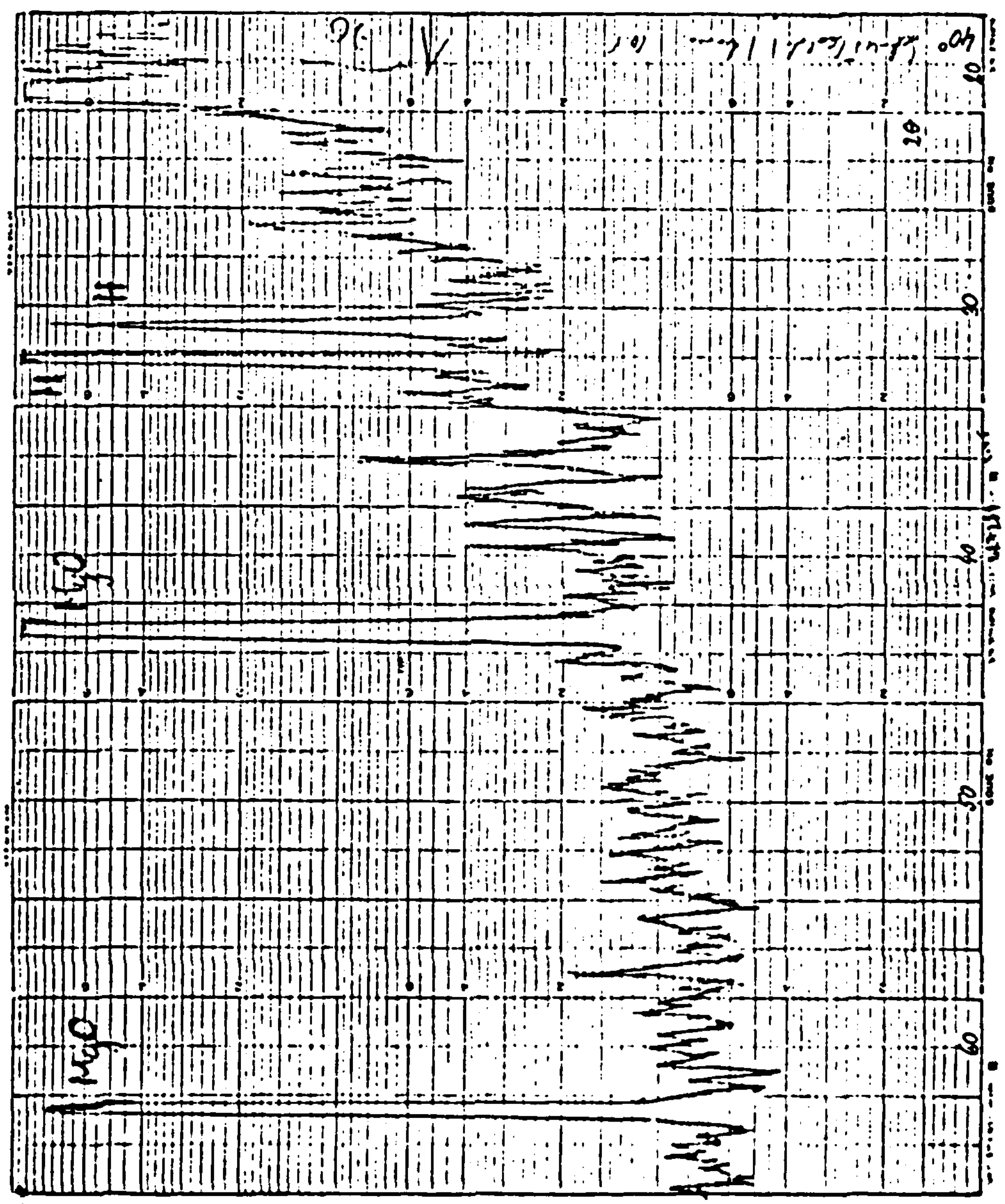


Fig. 33 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 100°F at 1 hour.

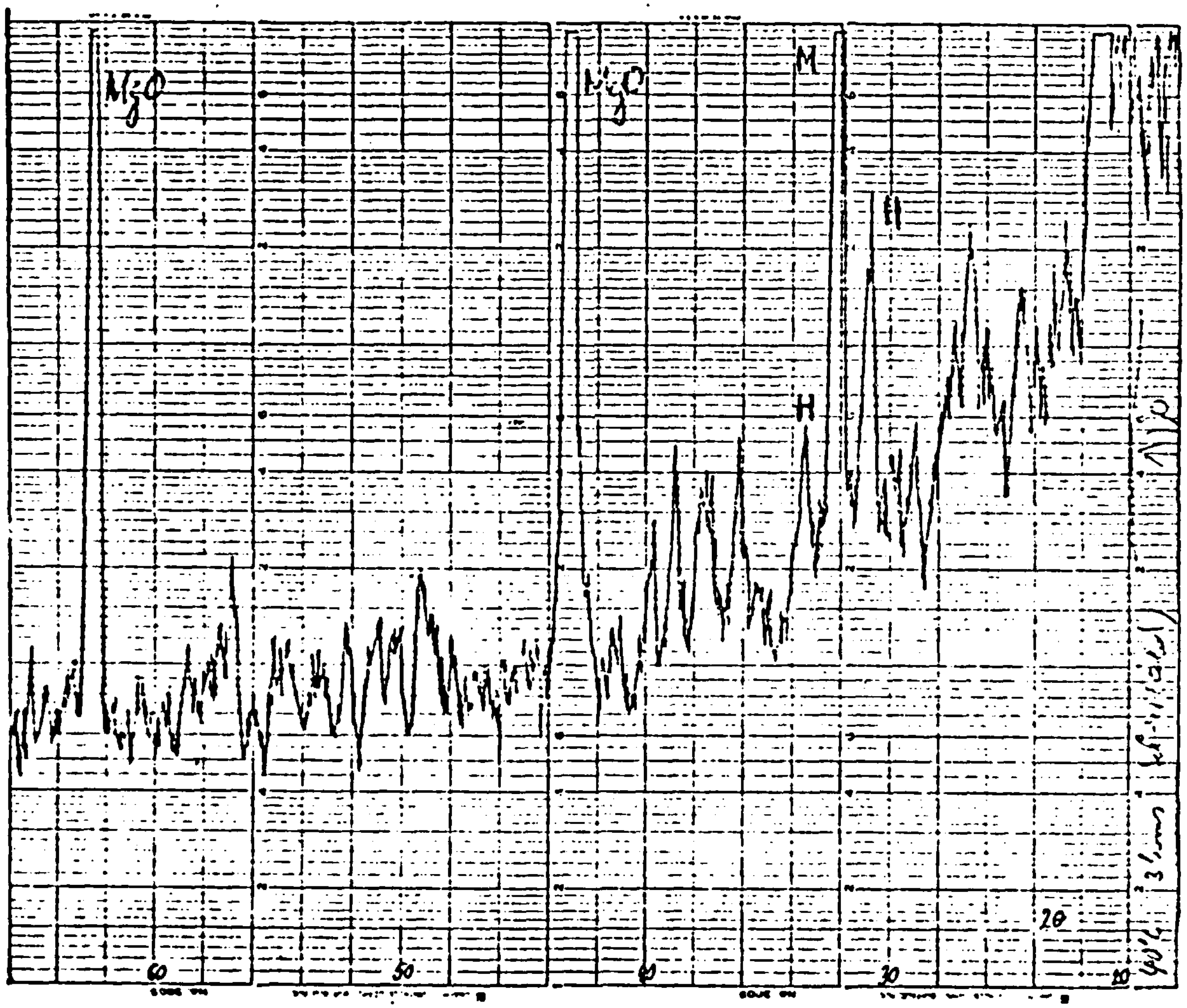


Fig. 34 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 100°F at 3 hours.

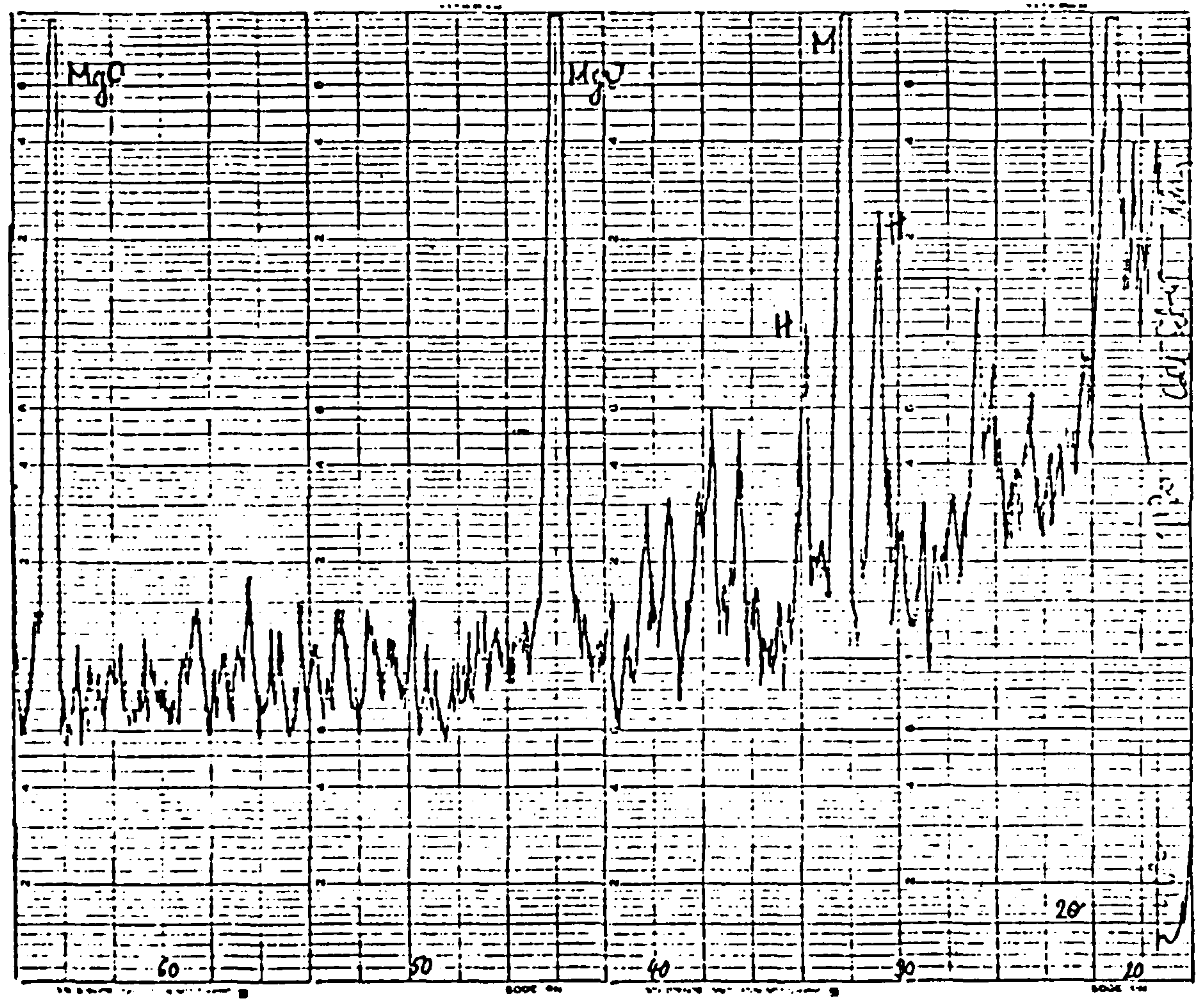


Fig. 35 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at

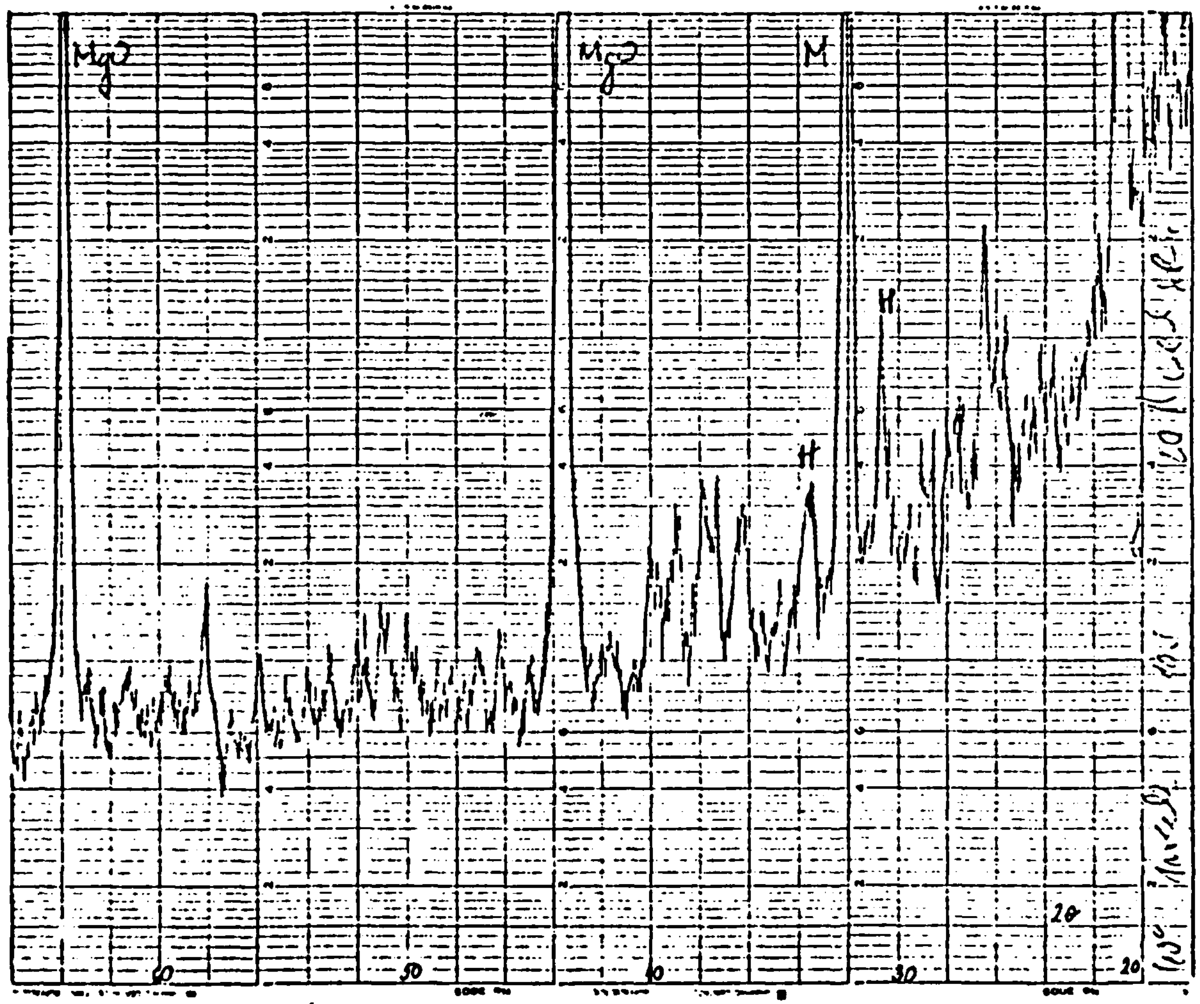


Fig. 36 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 100°F at [illegible] week.

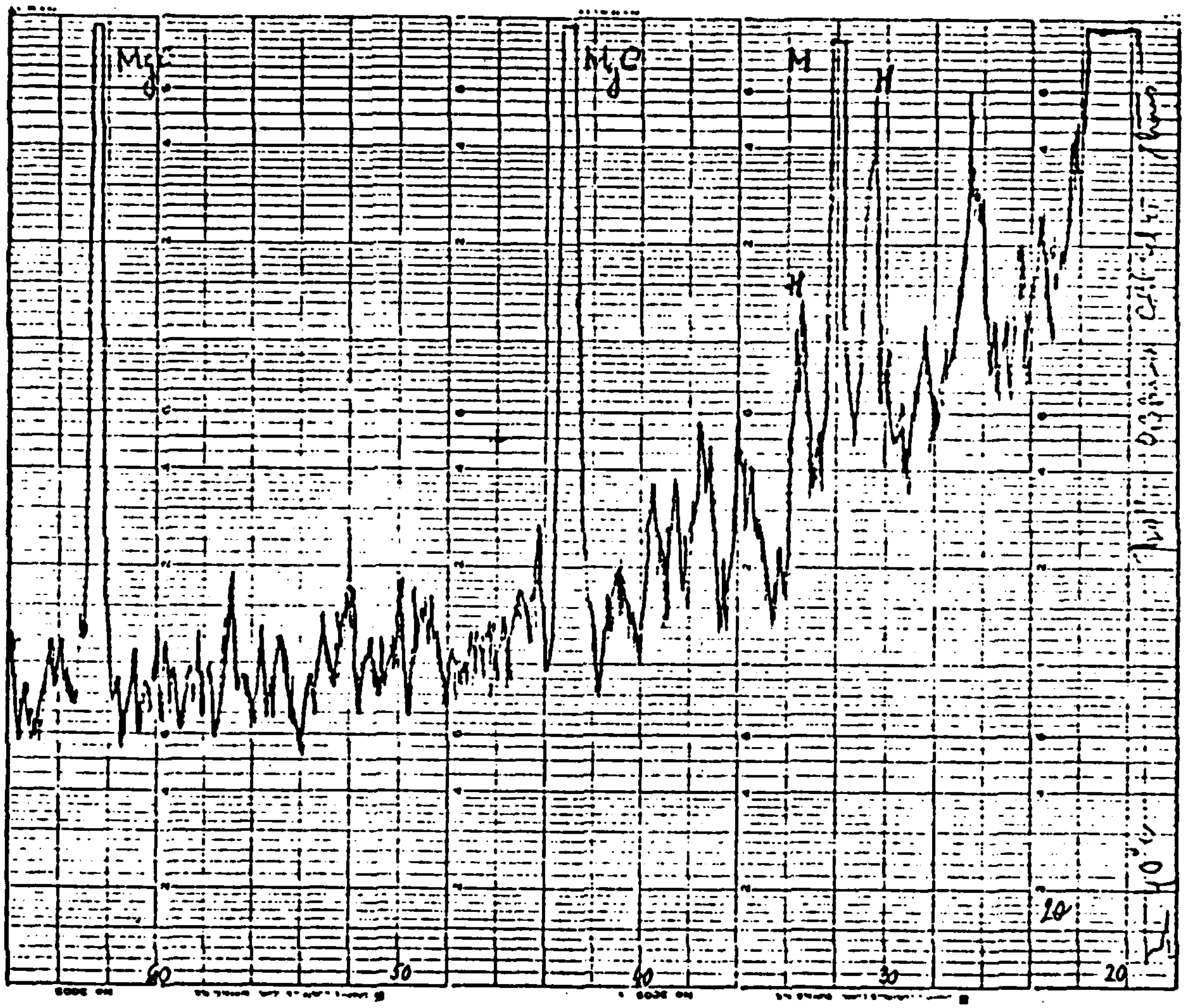


Fig. 37 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 100°F at 1 hour.

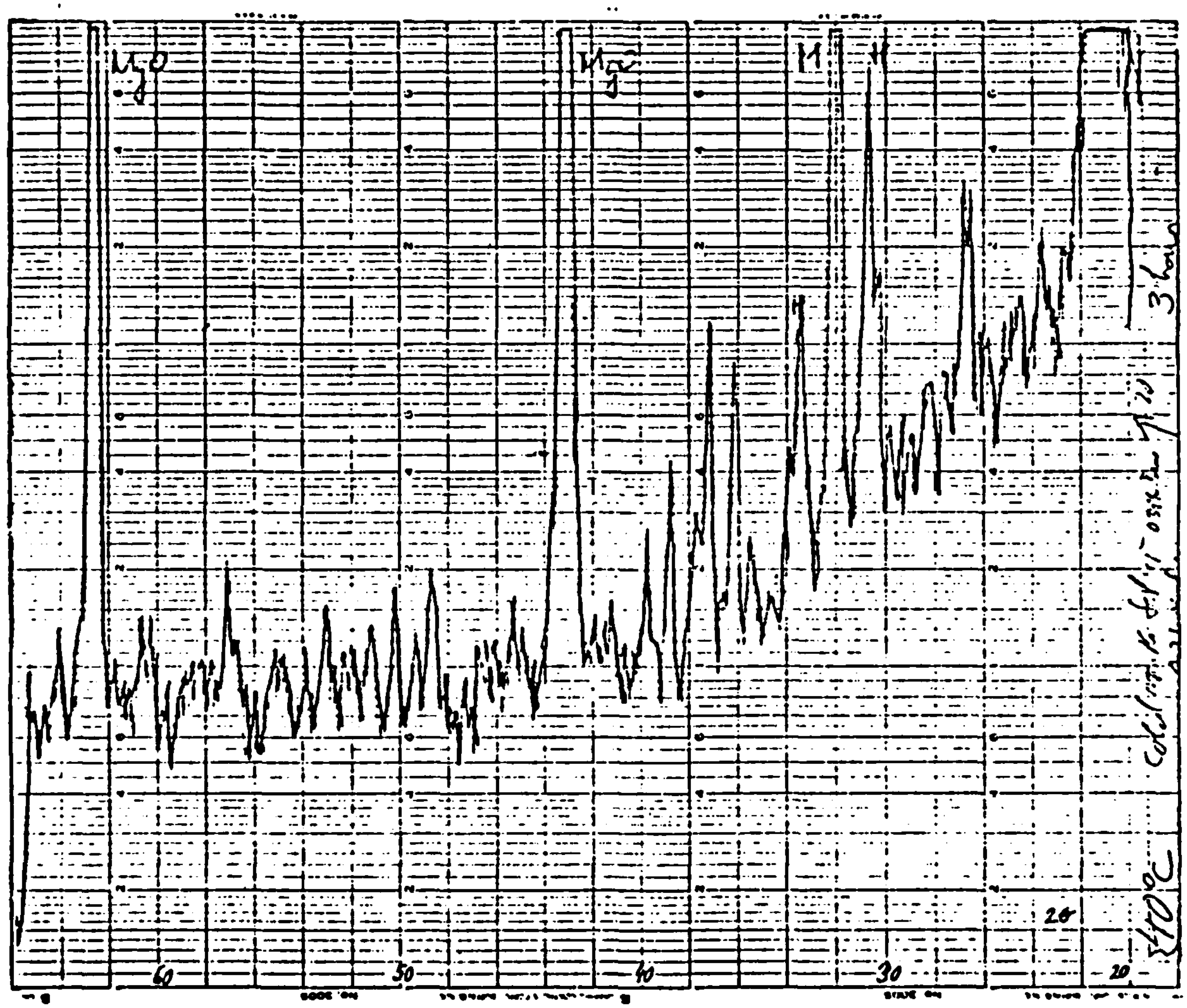


Fig. 38 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 100°F at 3 hours.

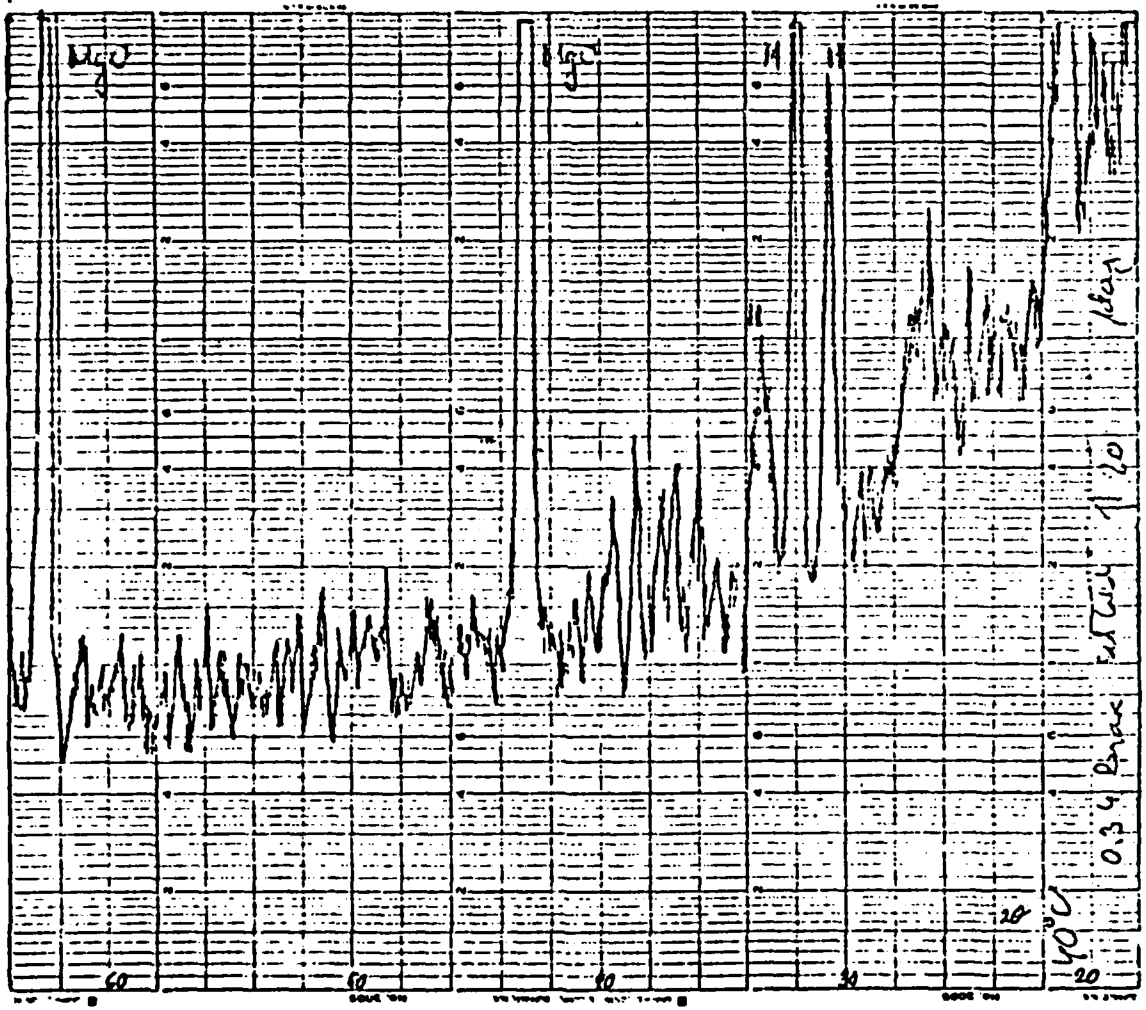


Fig. 39 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 100°F at 1 day.

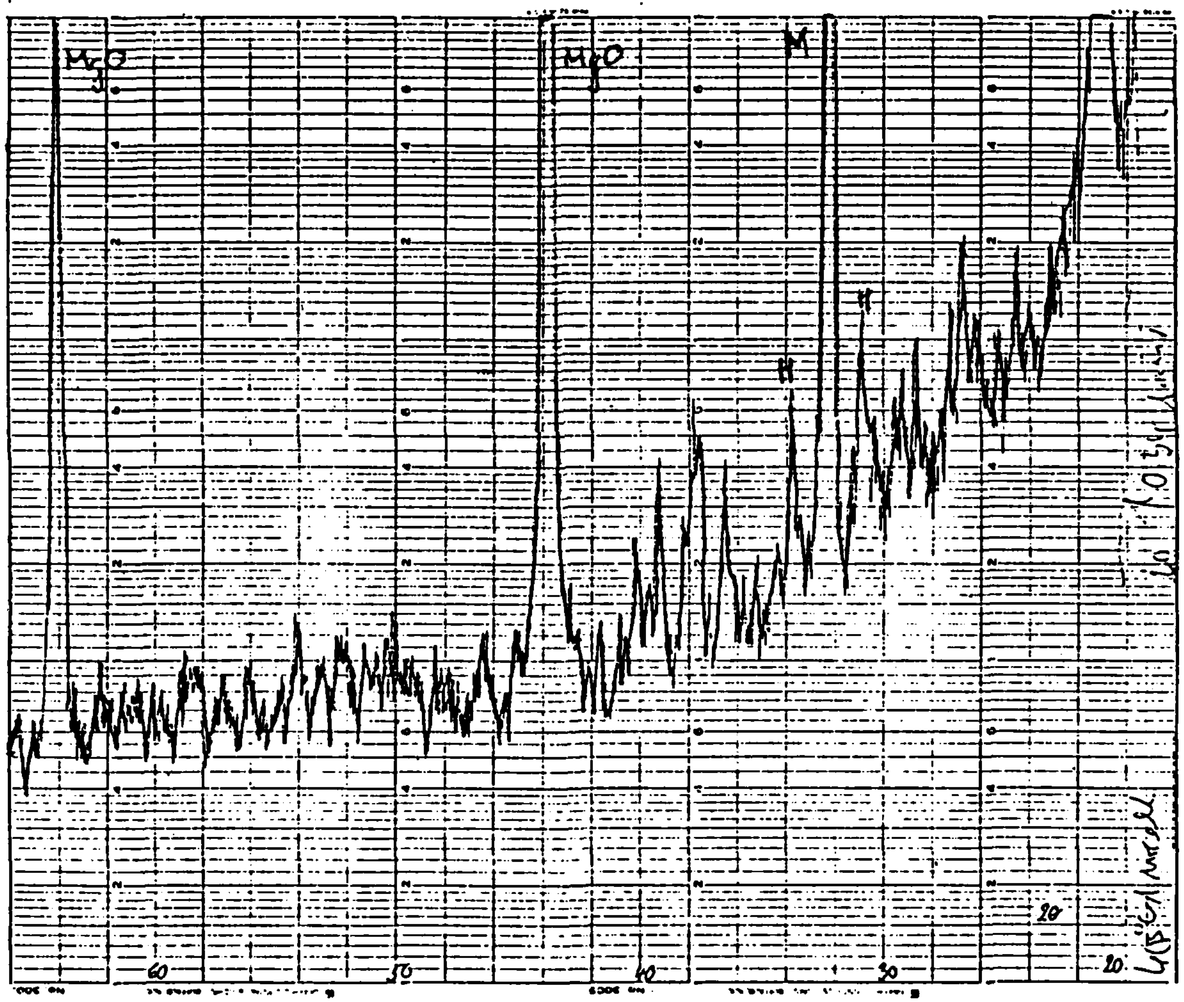


Fig. 40 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 100°F at 1 week.

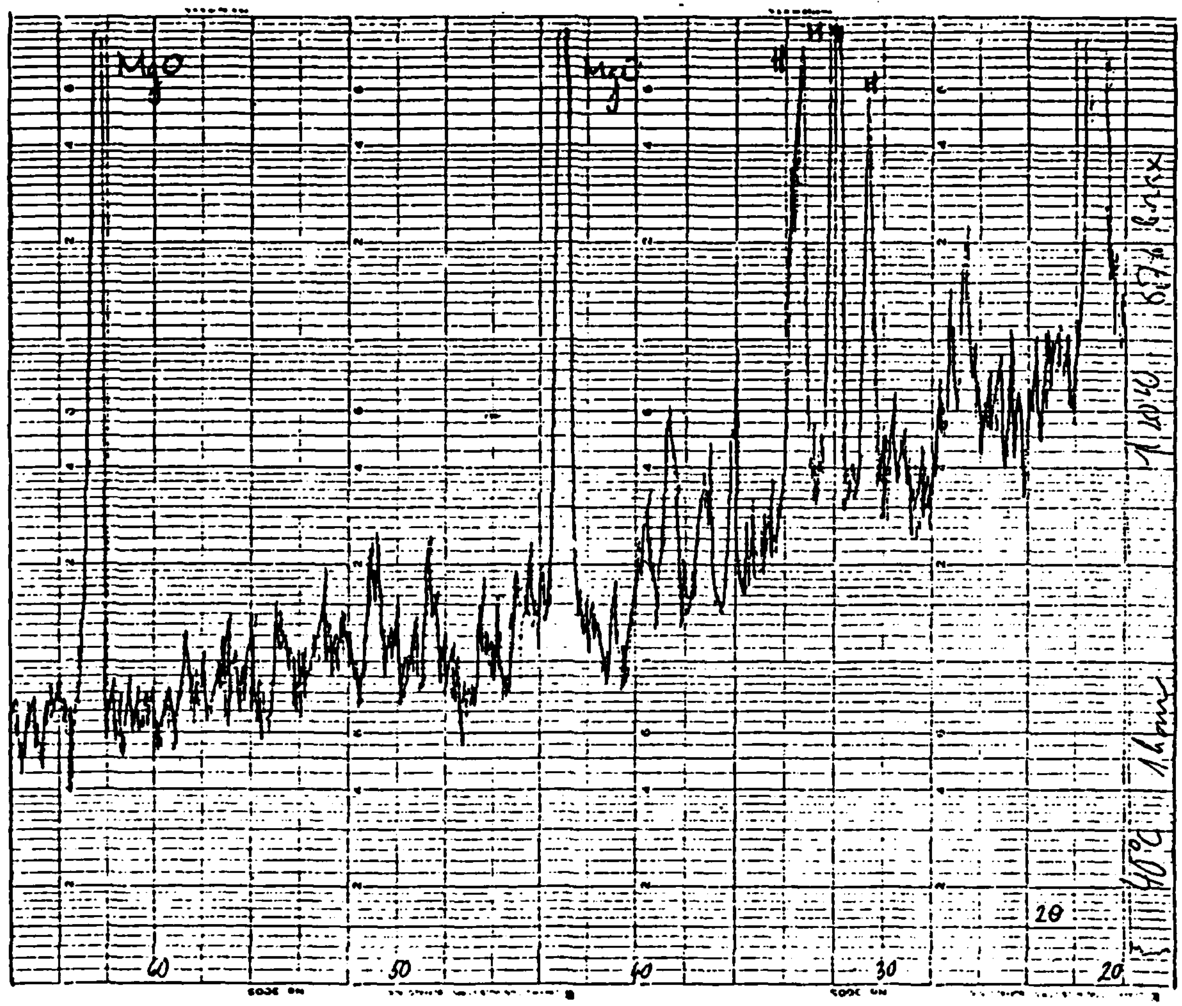


Fig. 41 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 100°F at 1 hour.

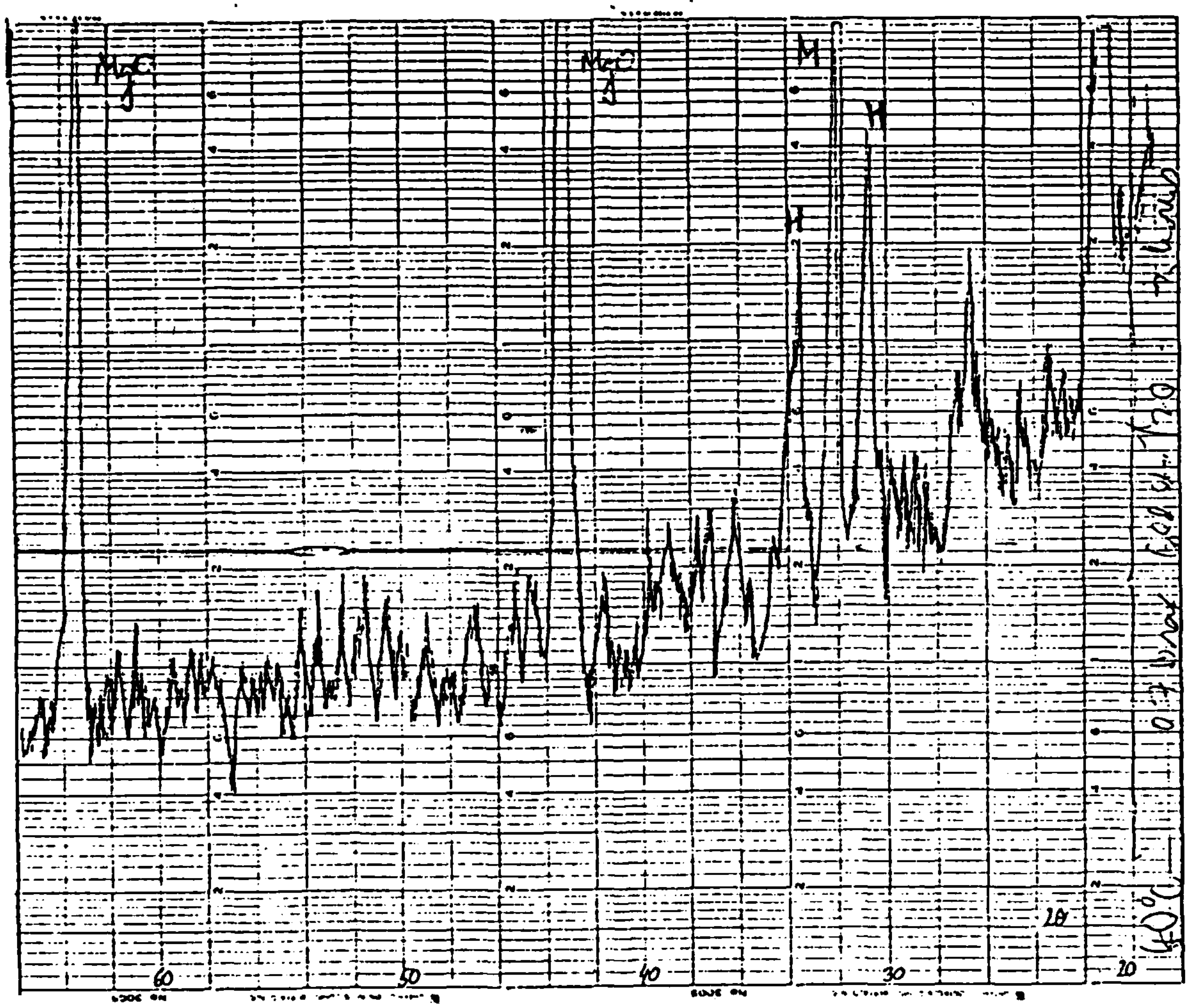


Fig. 42 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 100°F at 3 hours.

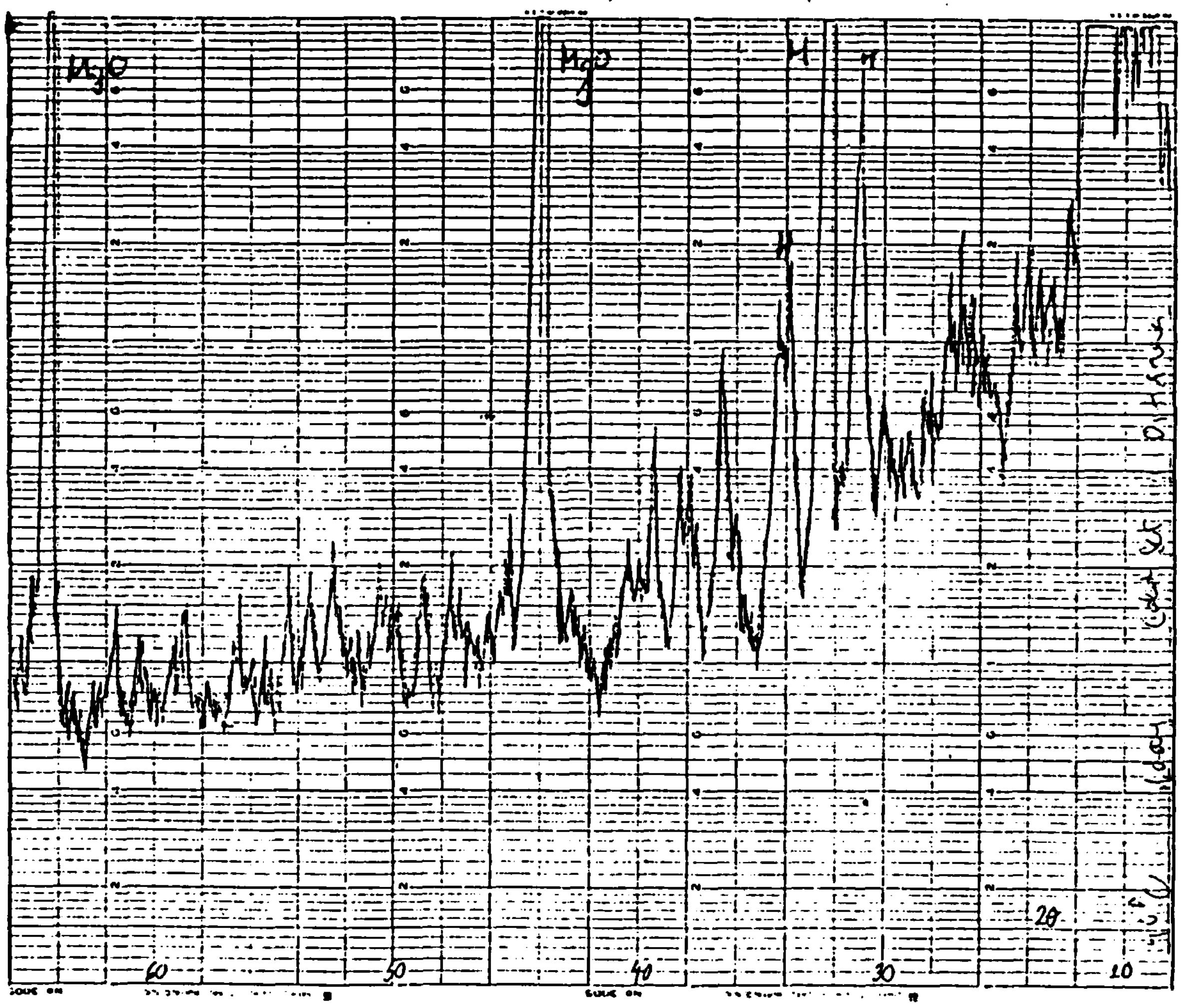


Fig. 43 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 100°F a 1 day.

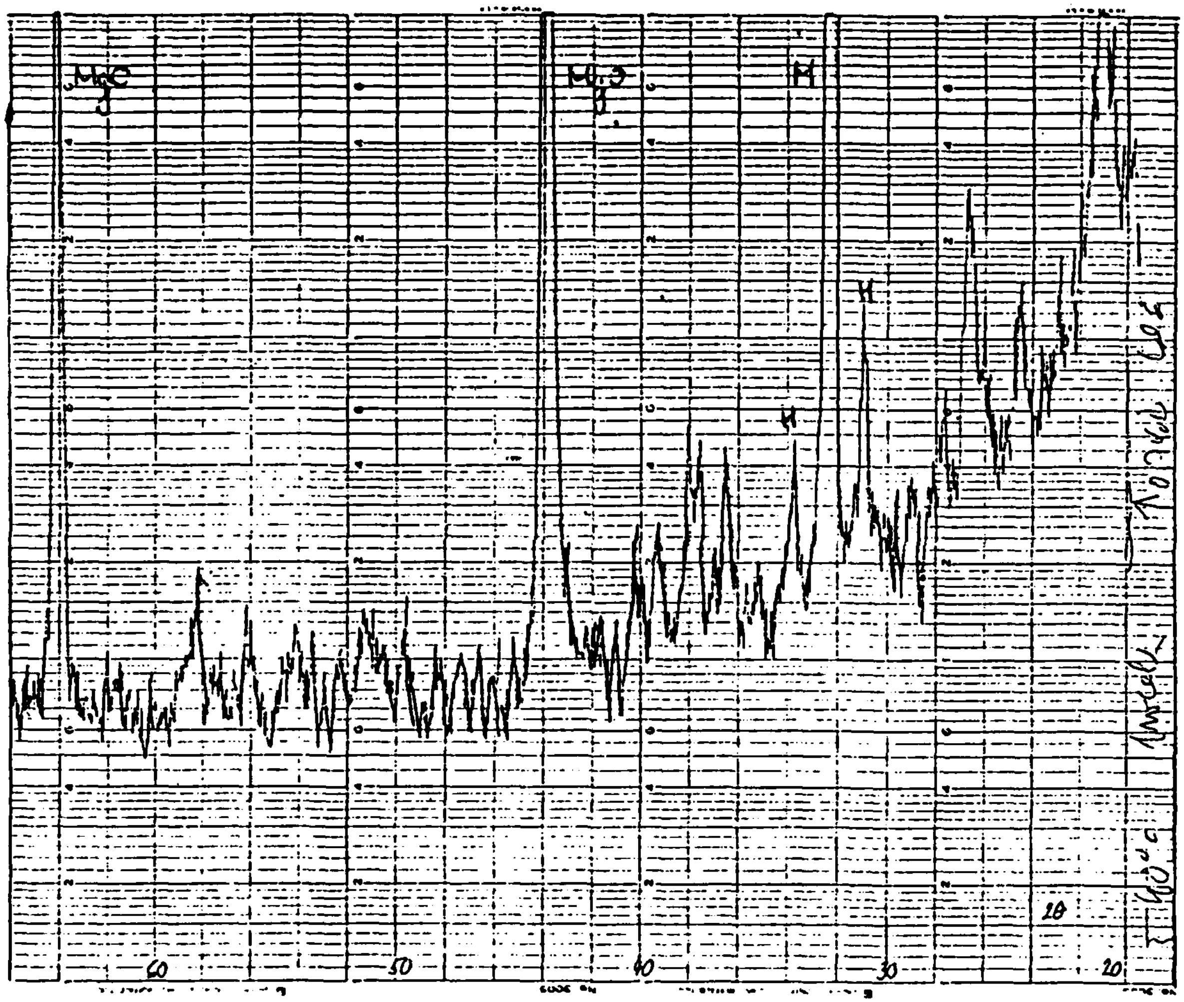


Fig. 44 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 100°F at 1 week.

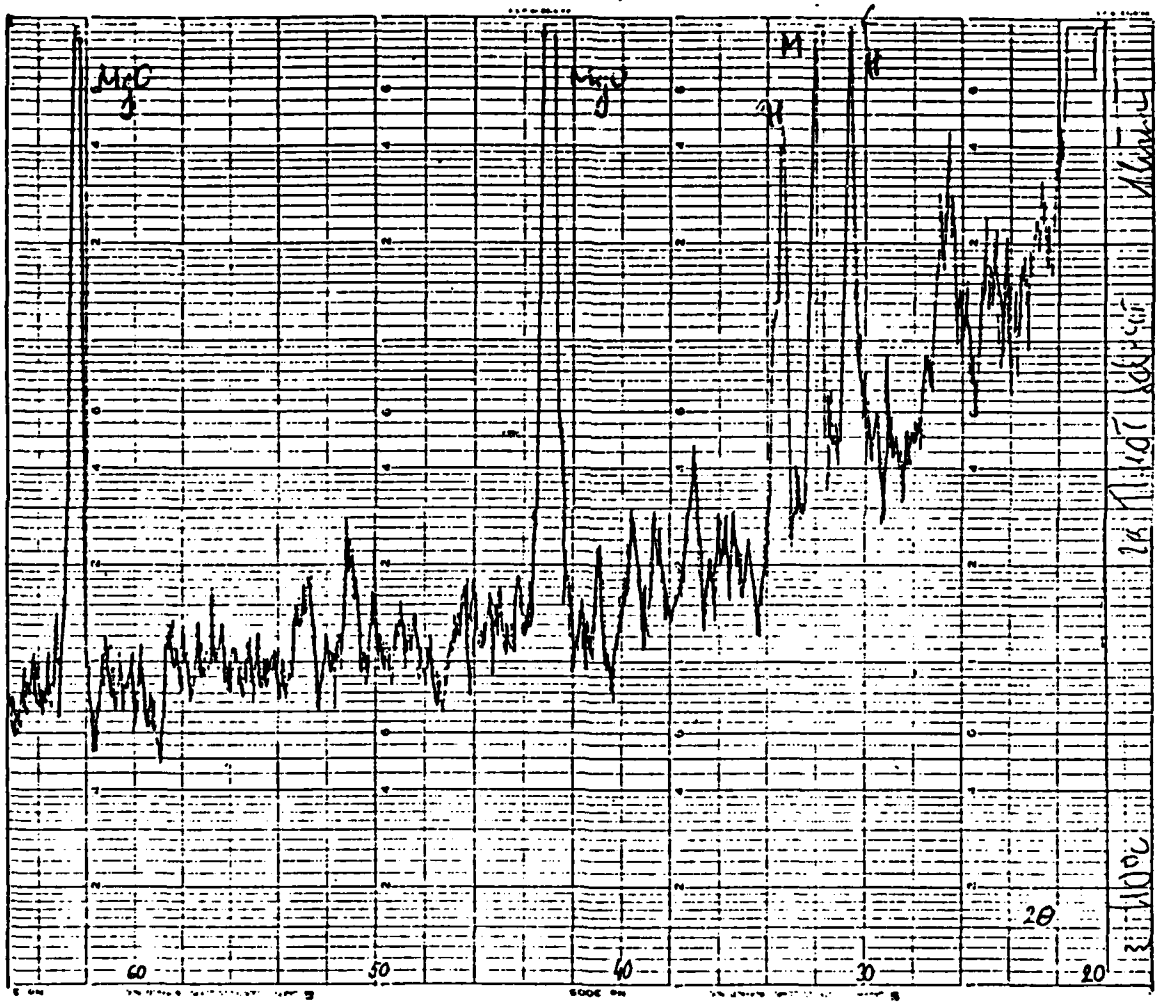


Fig. 45 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 100°F at 1 hour.

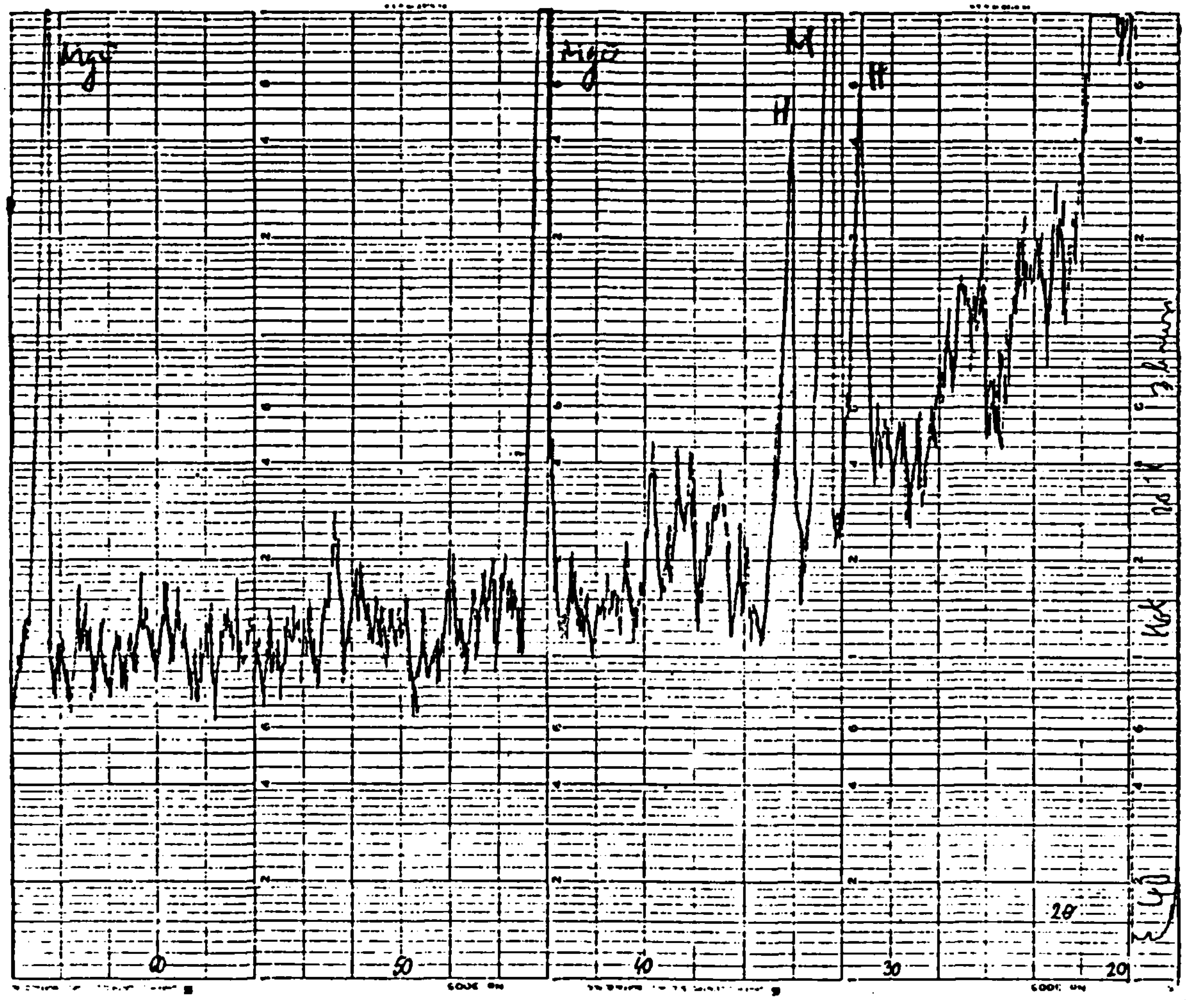


Fig. 46 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured
at 100°F at 3 hours.

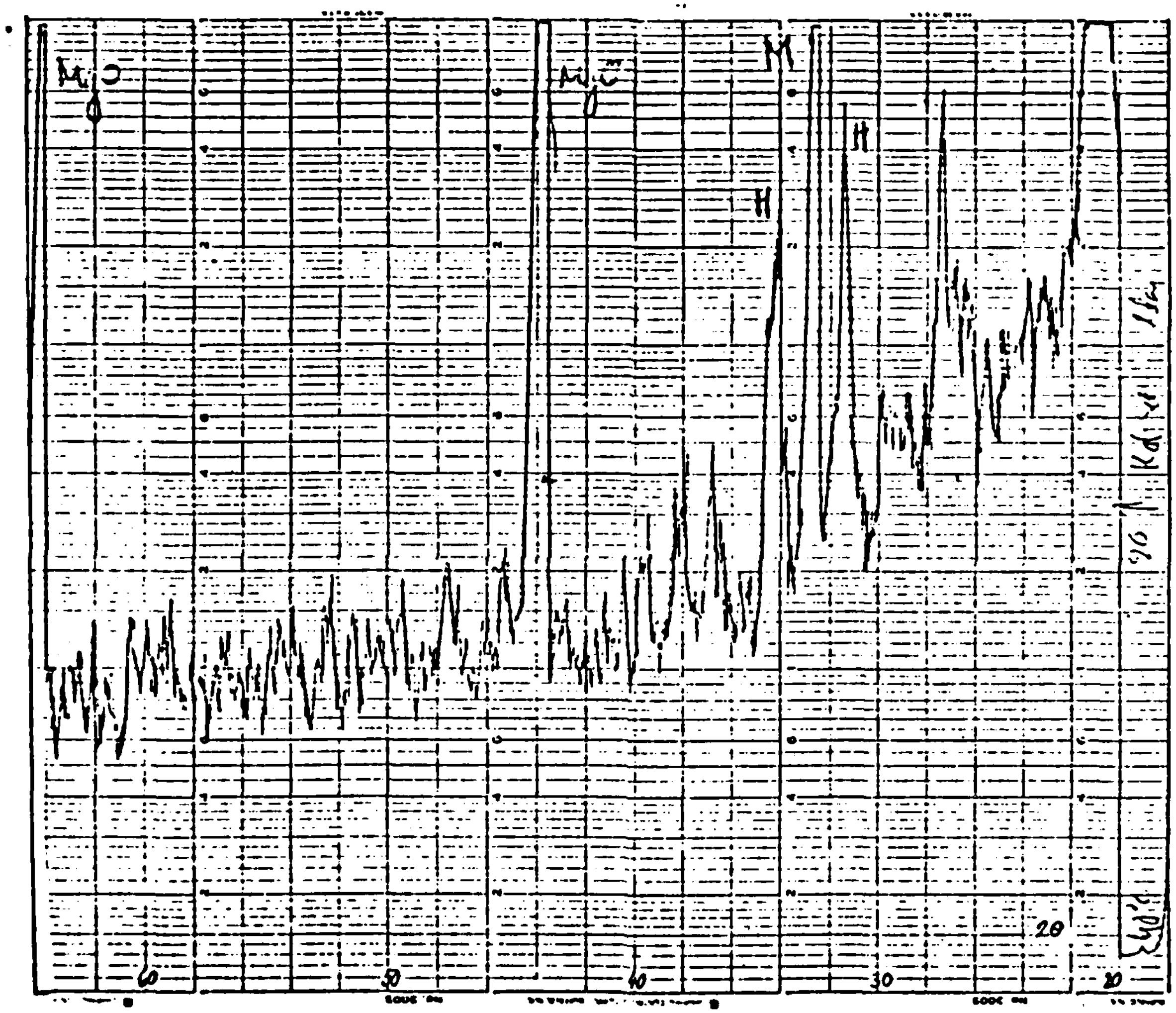


Fig. 47 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 100°F at 1 day.

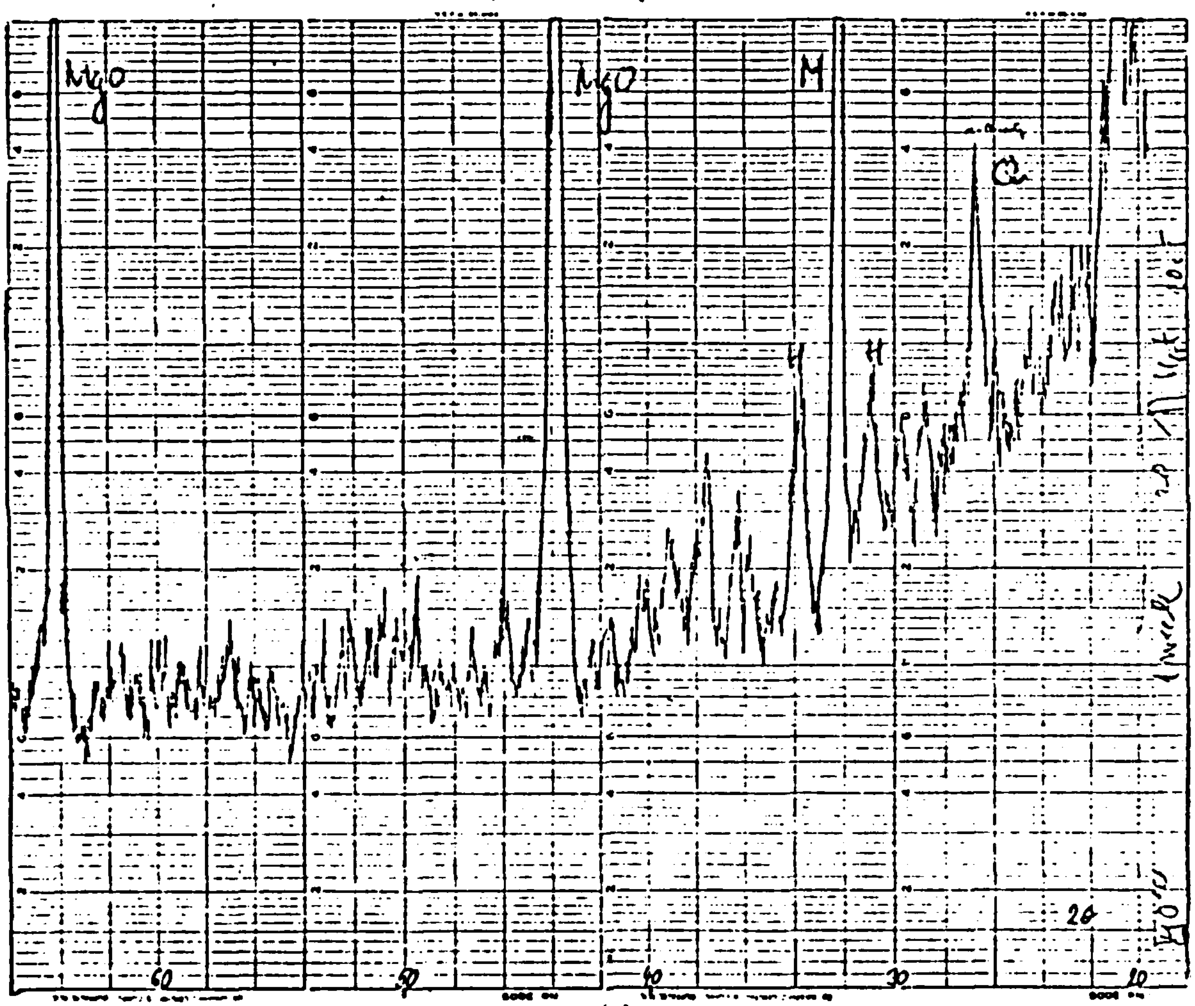


Fig. 48 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 100°F at 1 week.

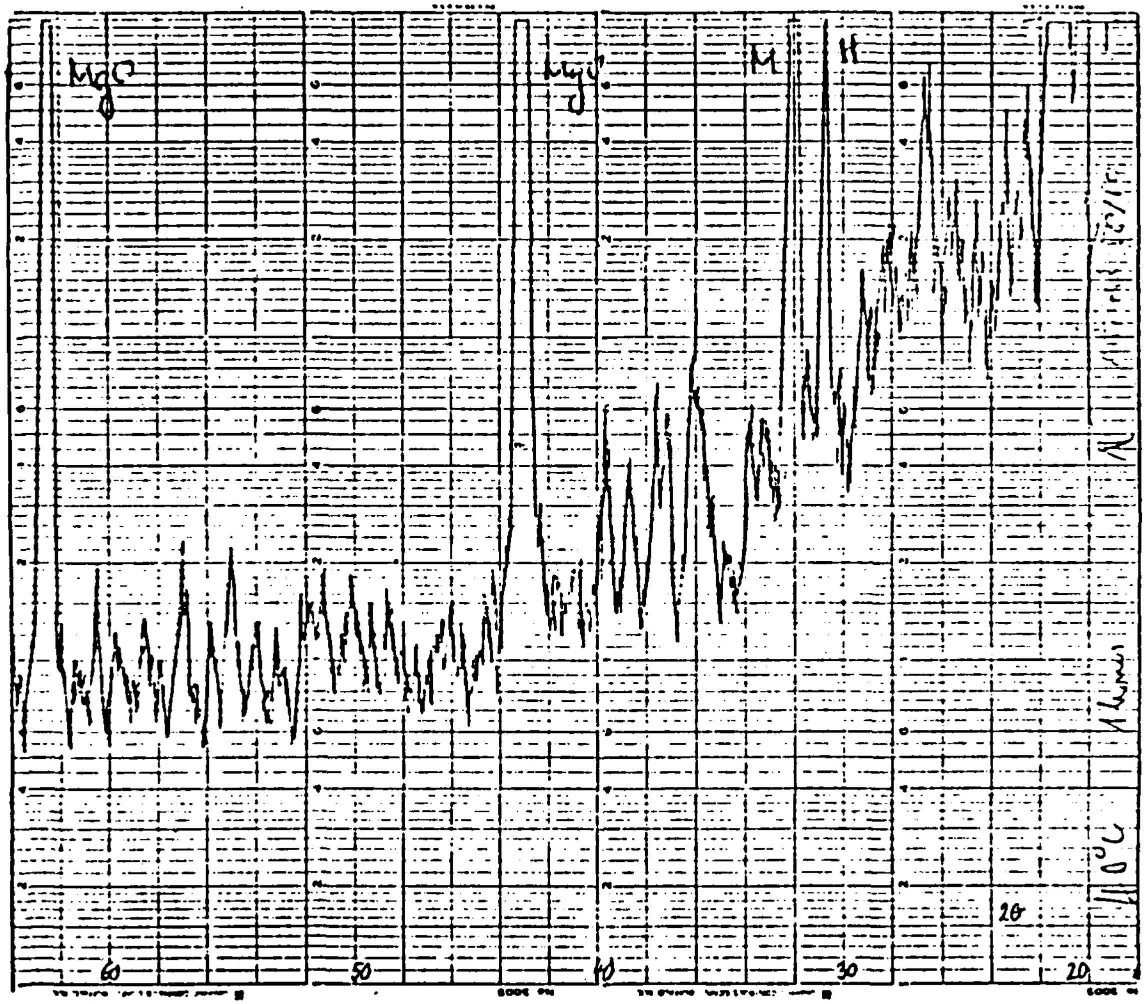


Fig. 49 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 100°F at 1 hour.

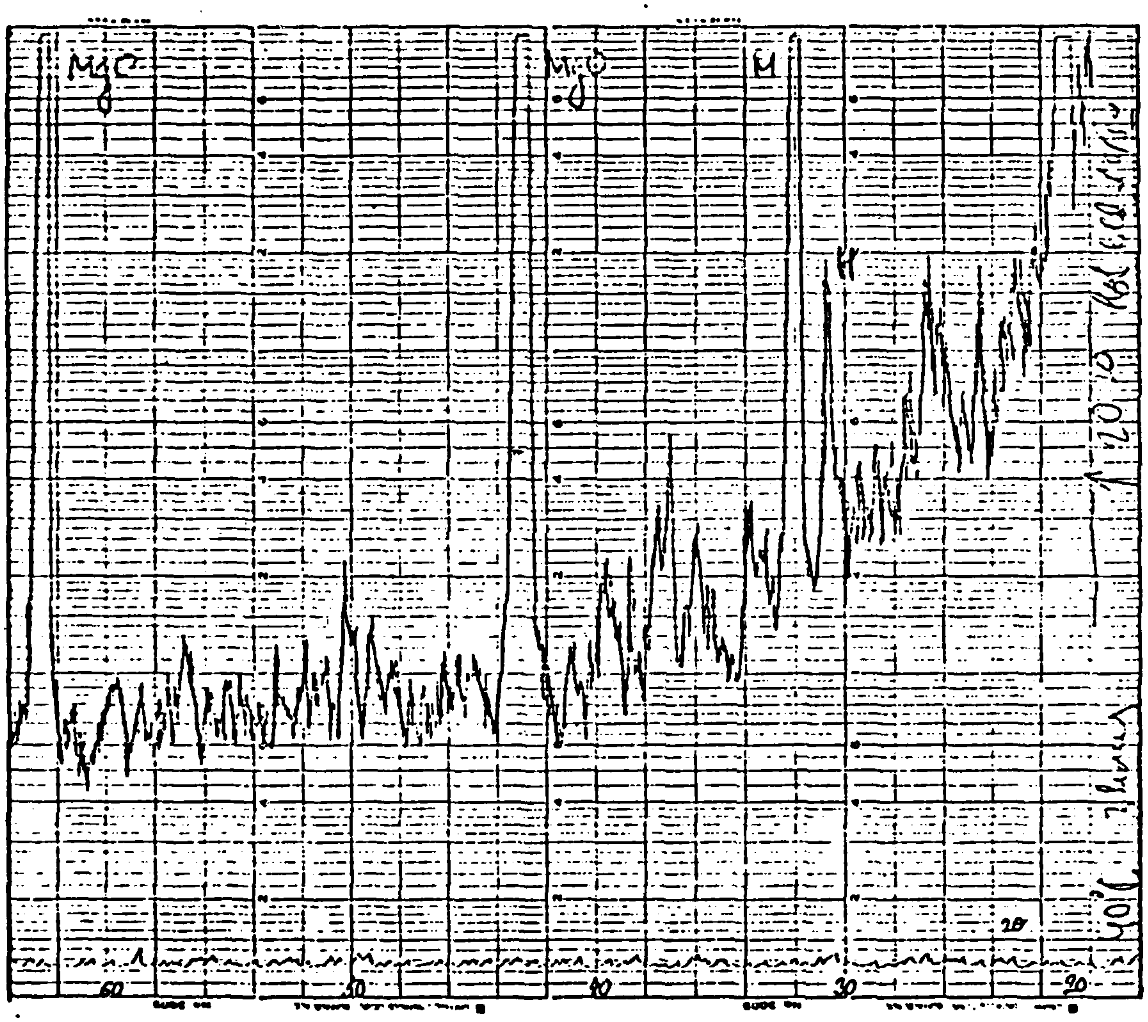


Fig. 50 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 100°F at 3 hours.

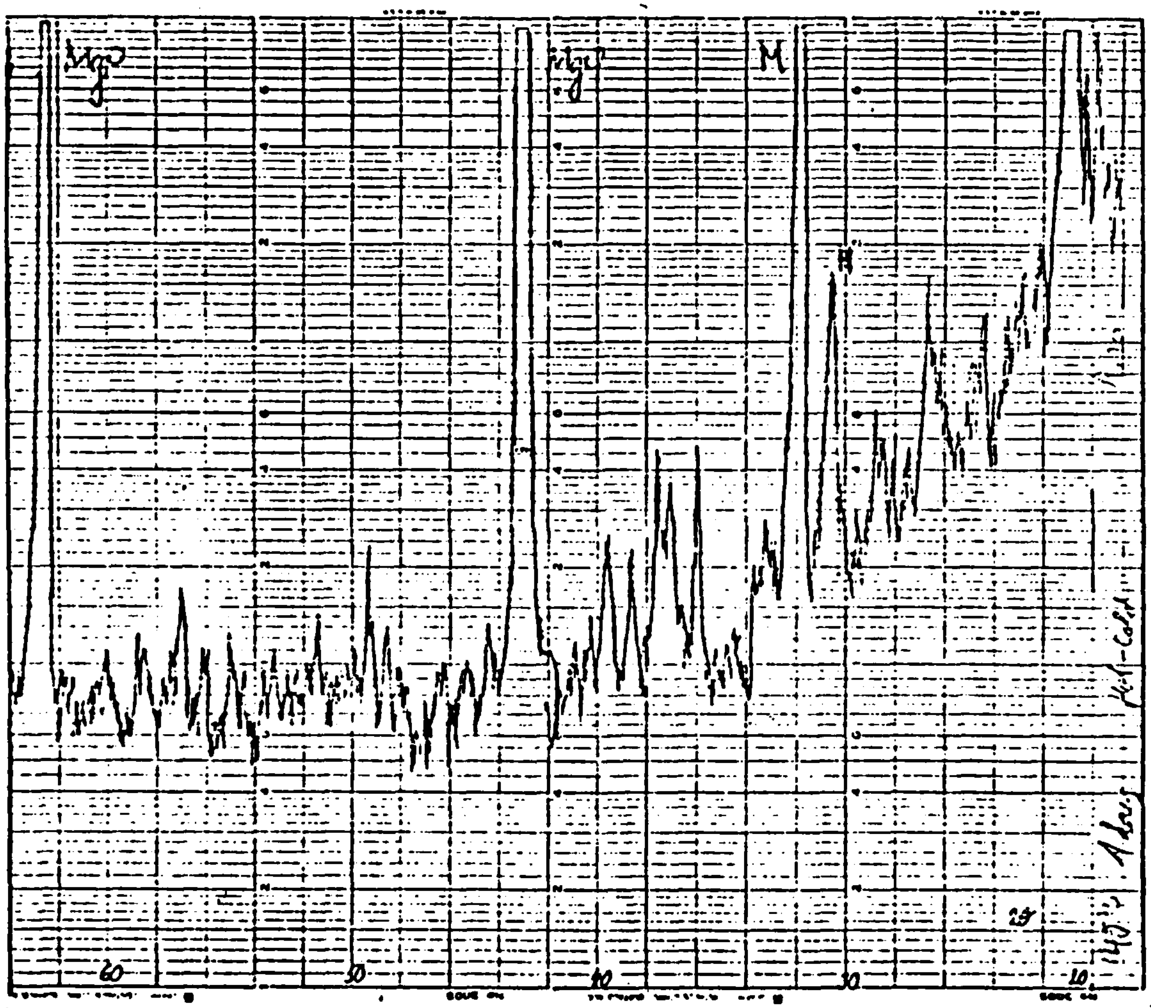


Fig. 51 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 100°F at [illegible] day.

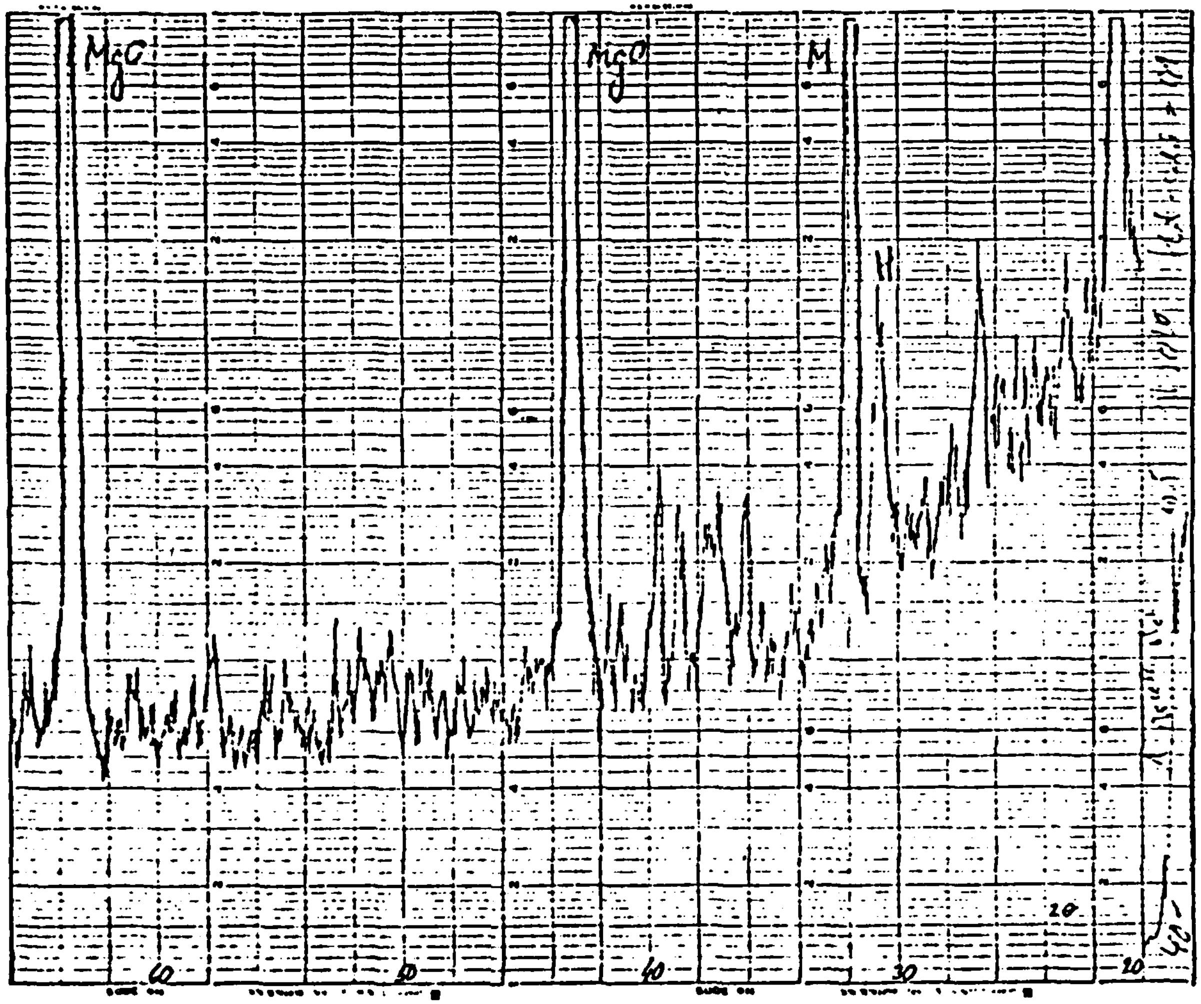


Fig. 52 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 100°F at 1 week.

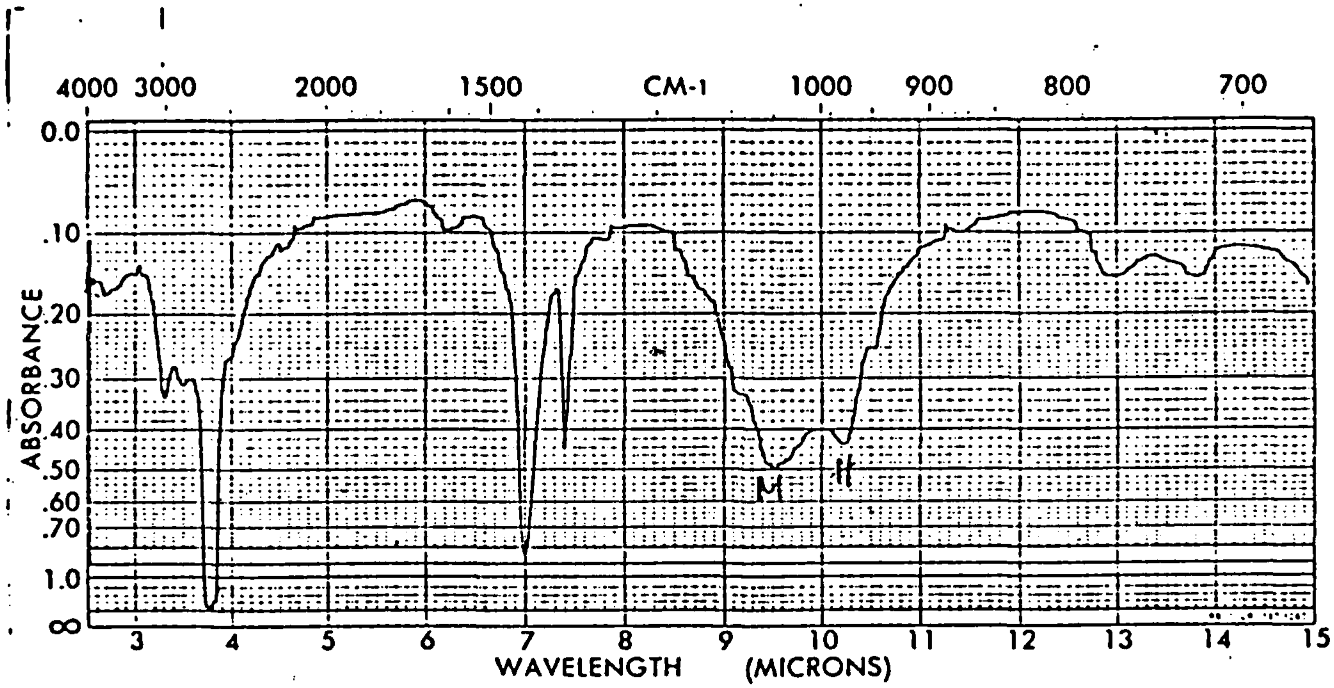


Fig. 53 - IR spectrum of SET-45 cold weather paste mixed and cured at 100°F at 3 days.

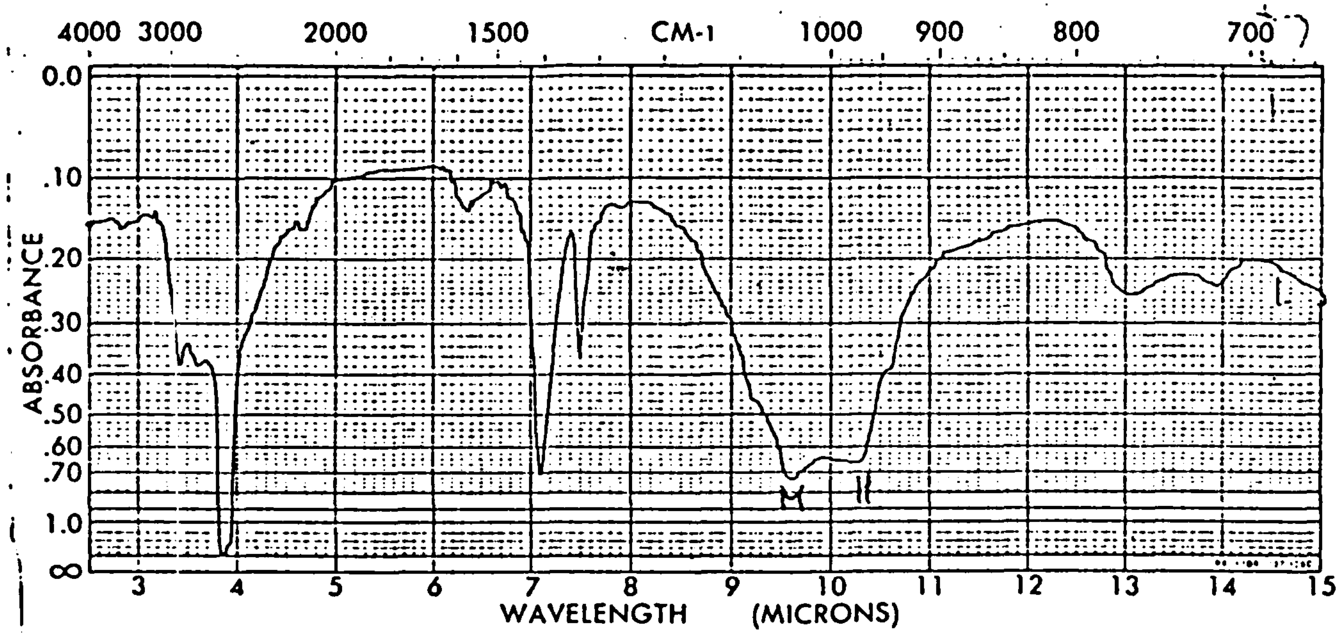


Fig. 54 - IR spectrum of SET-45 cold weather paste with 1.75% borax mixed and cured at 100°F at 3 days.

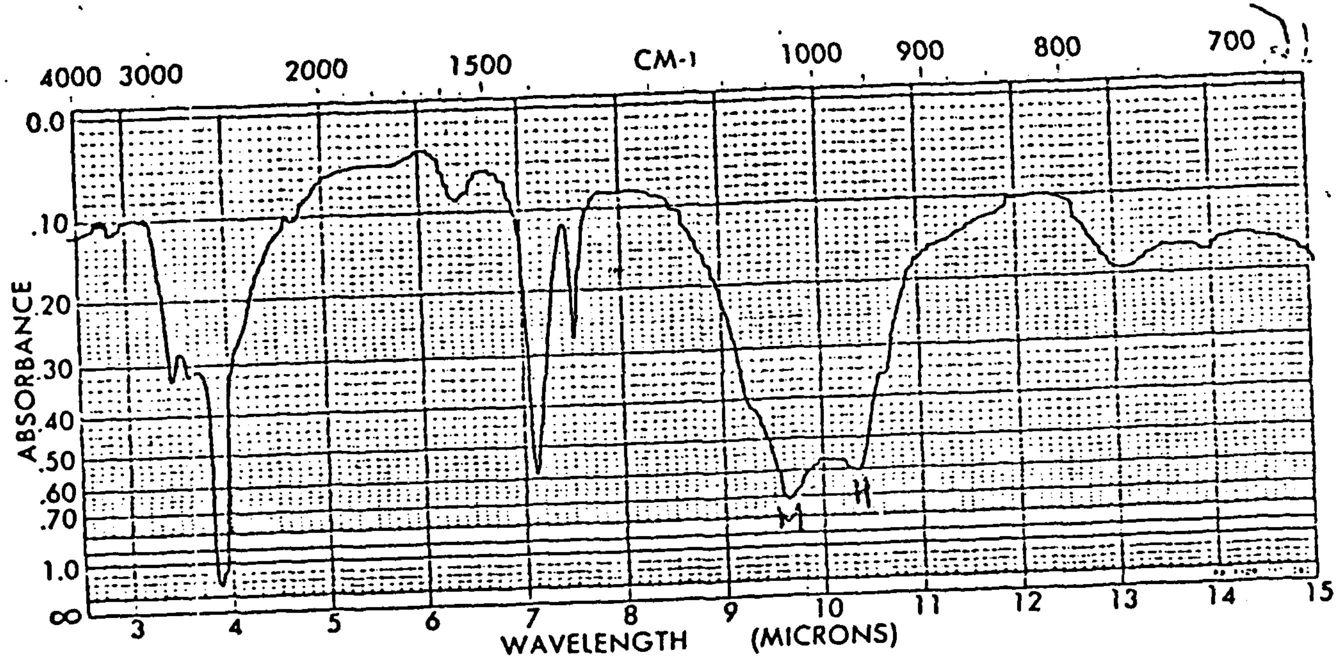


Fig. 55 - IR spectrum of SET-45 cold weather paste with 3.5% borax mixed and cured at 100°F at 3 days.

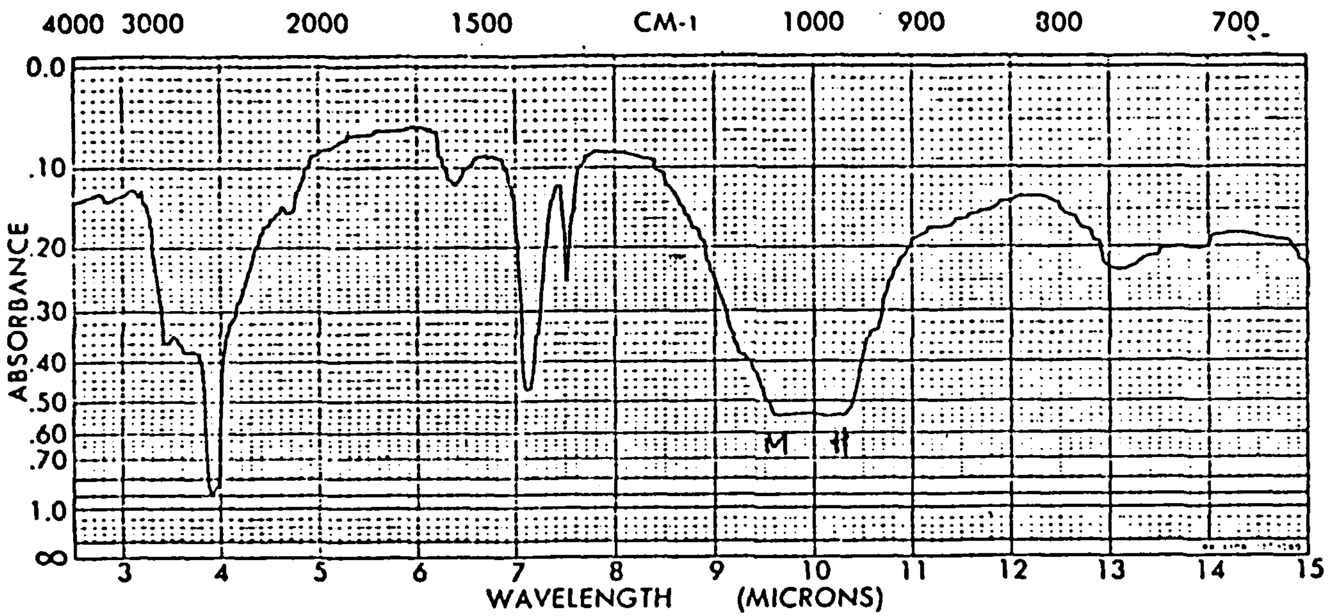


Fig. 56 - IR spectrum of SET-45 hot weather paste mixed and cured at 100°F at 3 days.

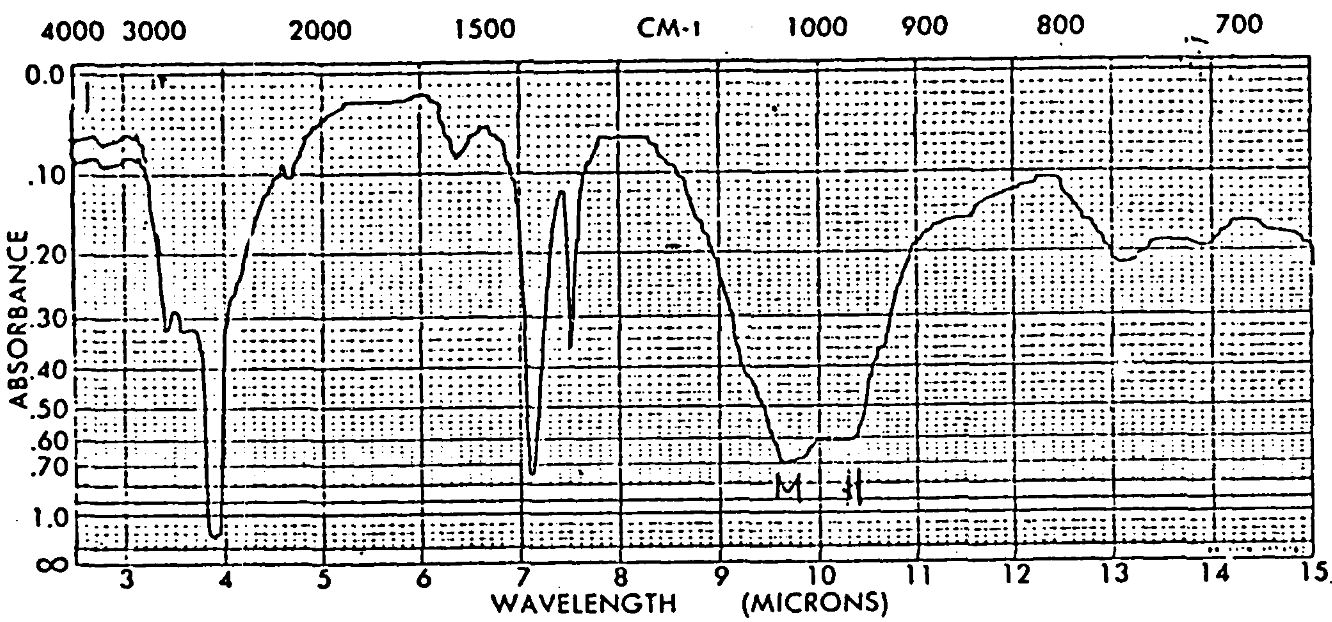


Fig. 57 - IR spectrum at SET-45 cold:hot = 1:1 paste mixed and cured at 100°F at 3 days.

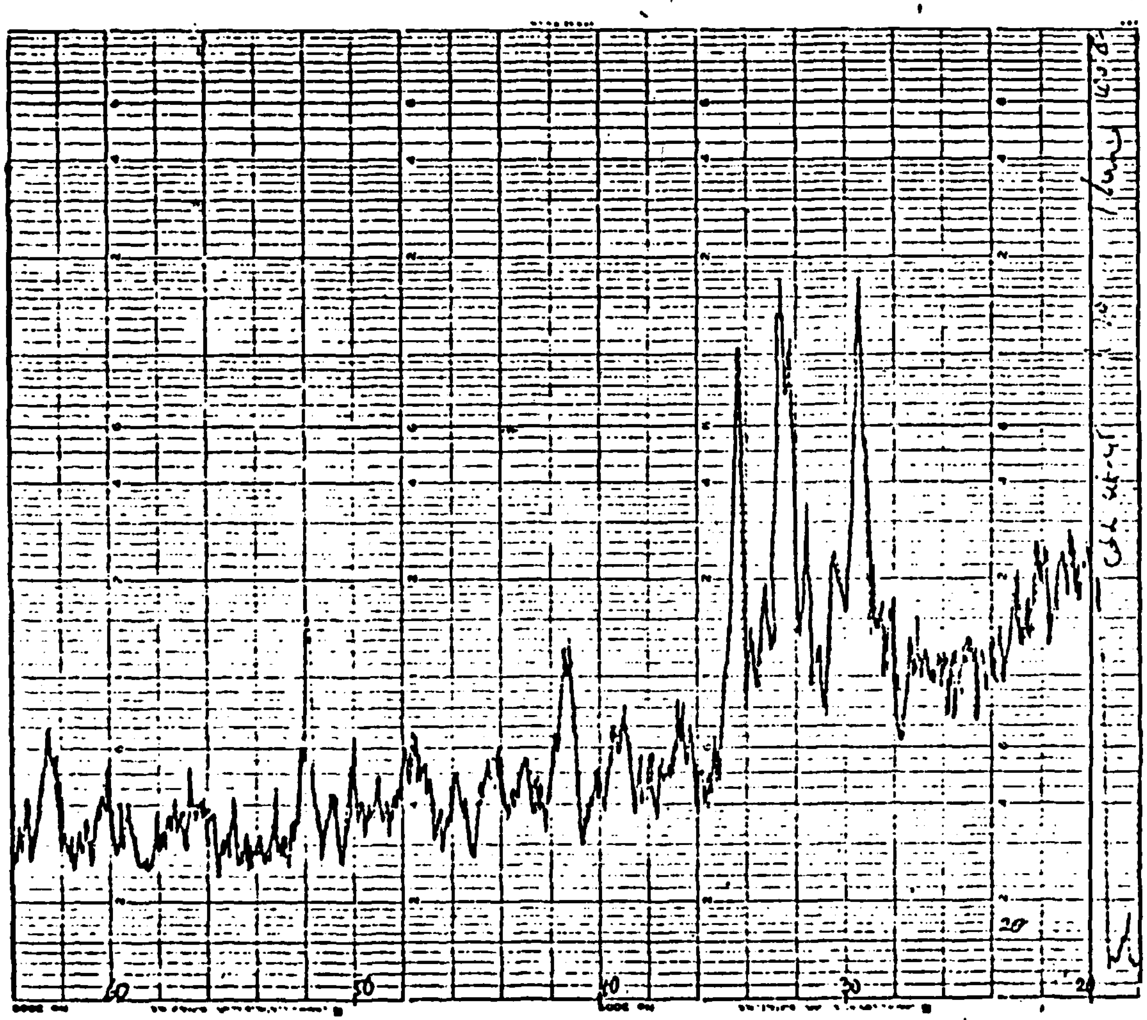


Fig. 58 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 200°F at 1 hour.

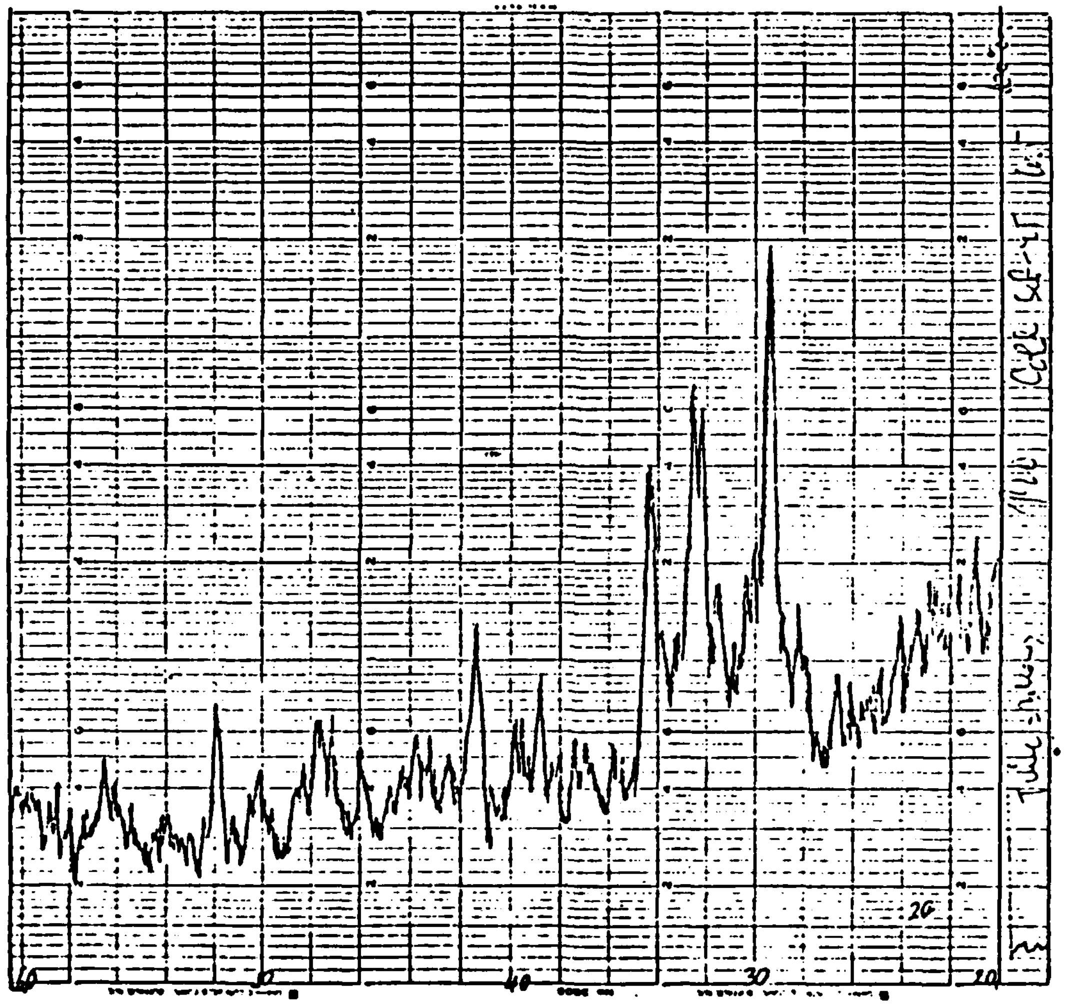


Fig. 59 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 200°F at 3 hours.

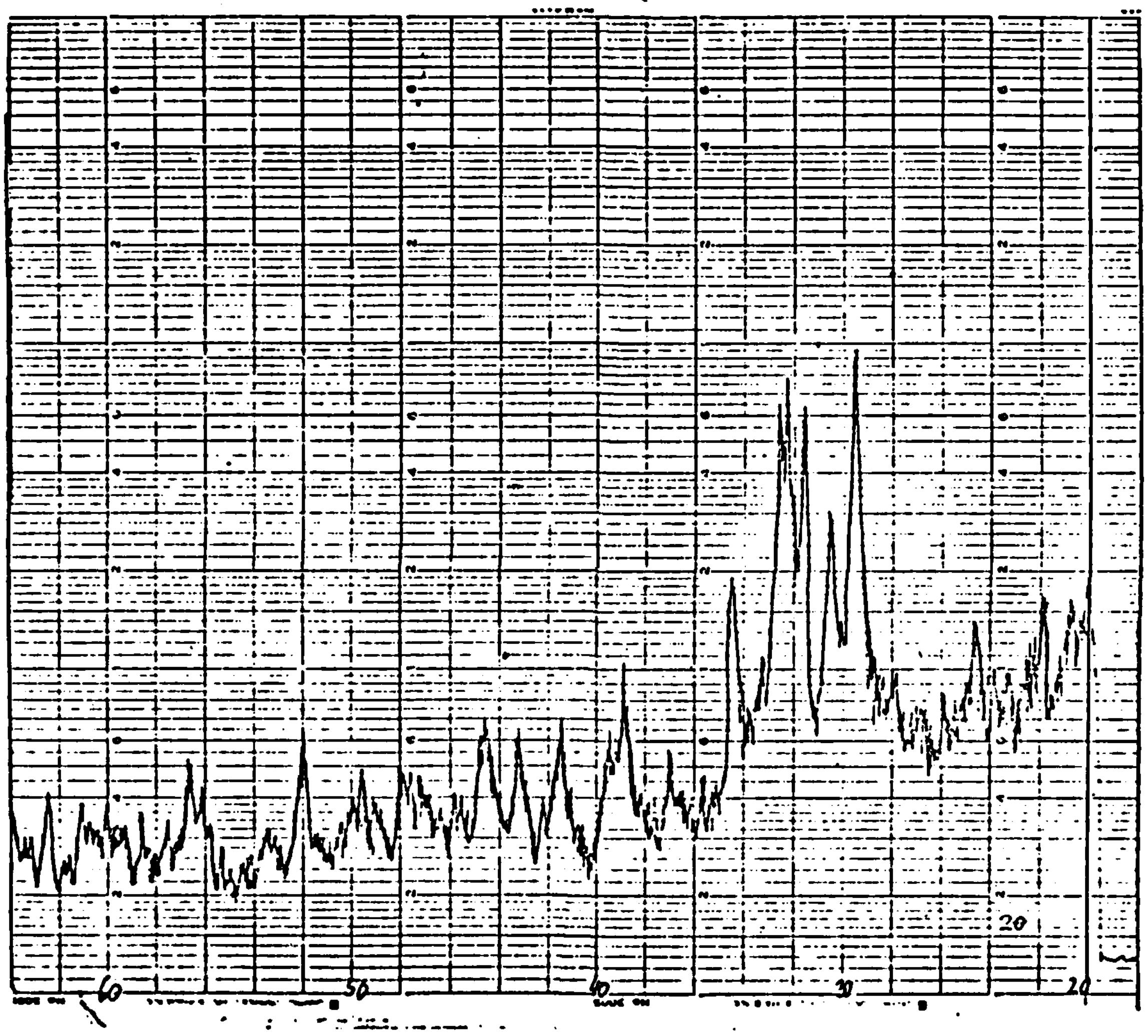


Fig. 60 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 200°F at 1 day.

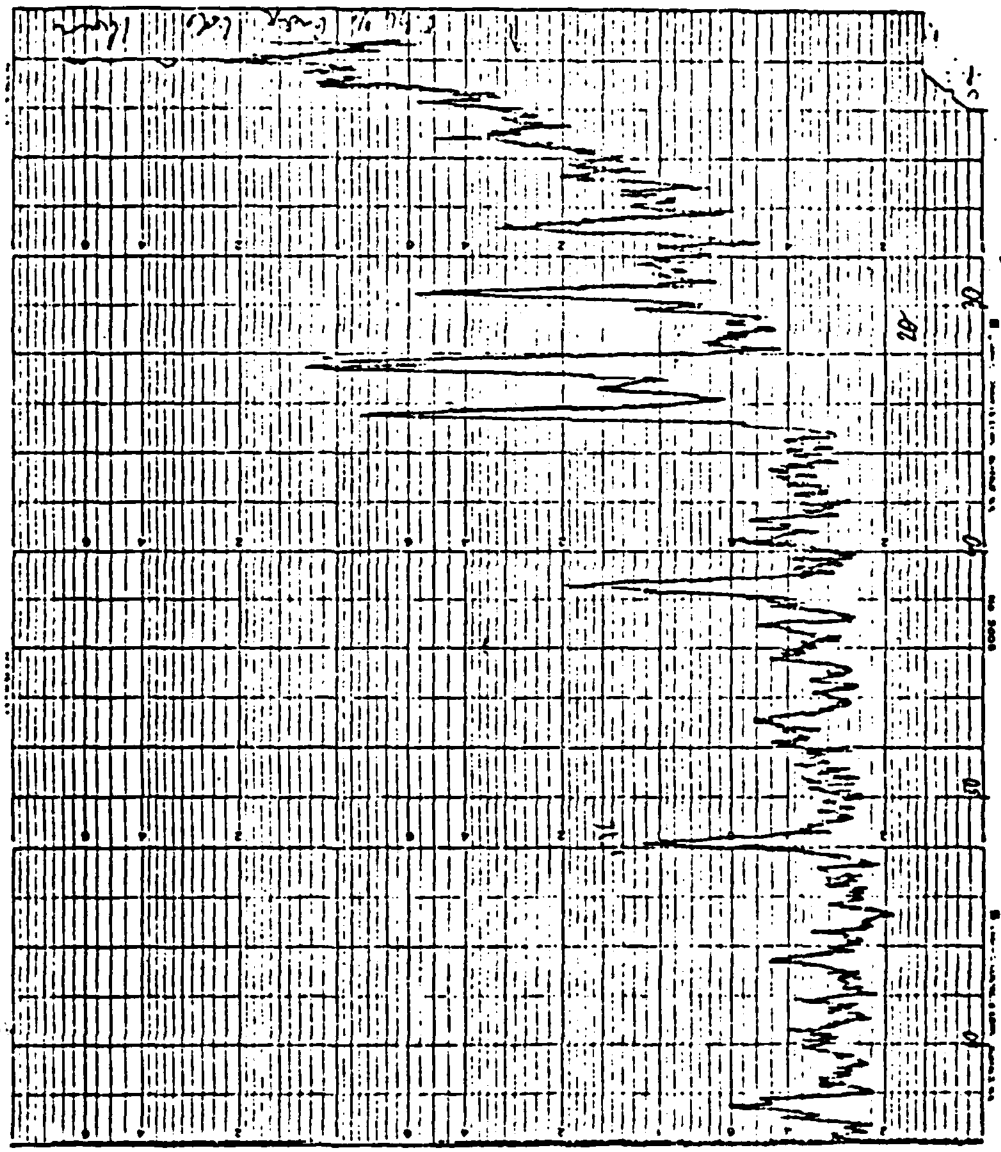


Fig. 61 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 200°F at 1 hour.

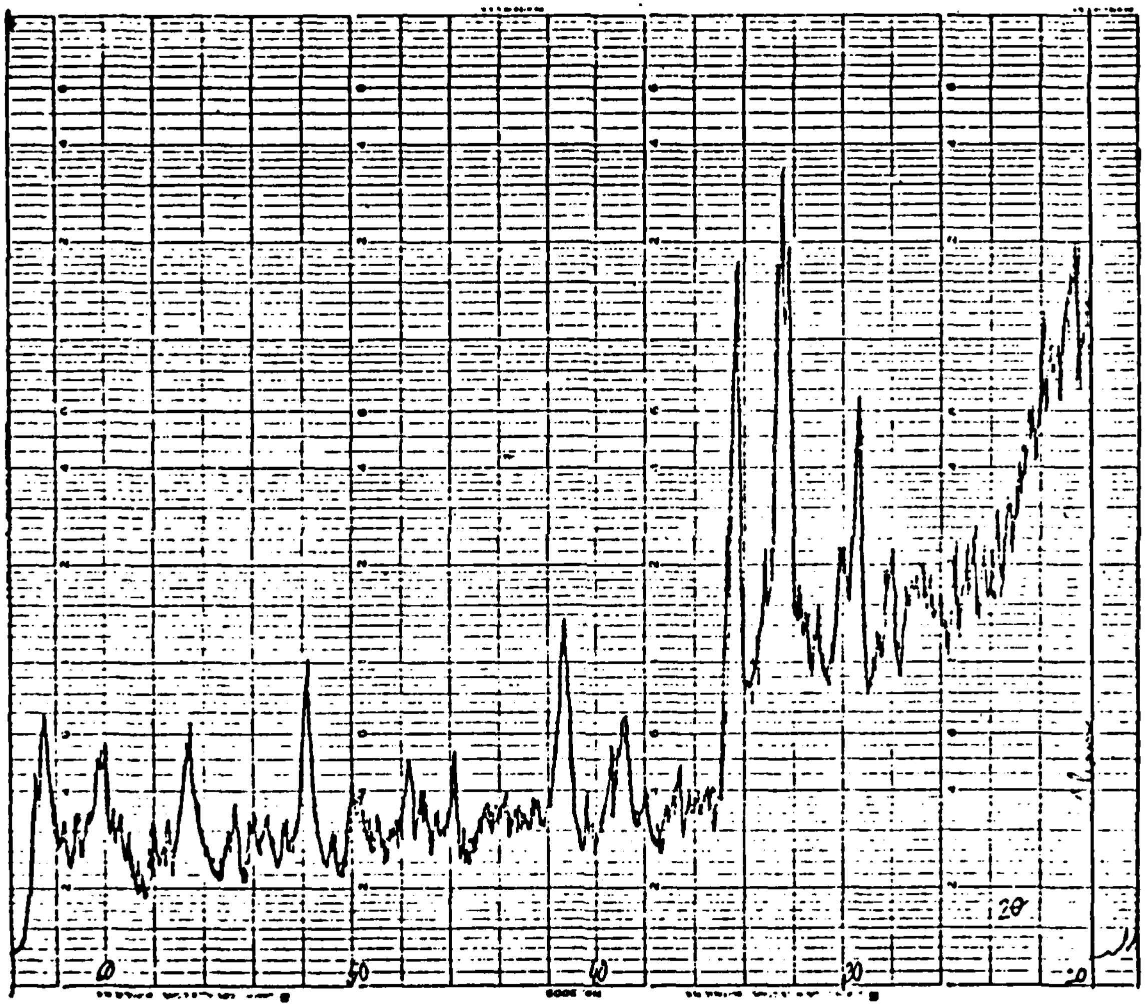


Fig. 62 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 200°F at 3 hours.

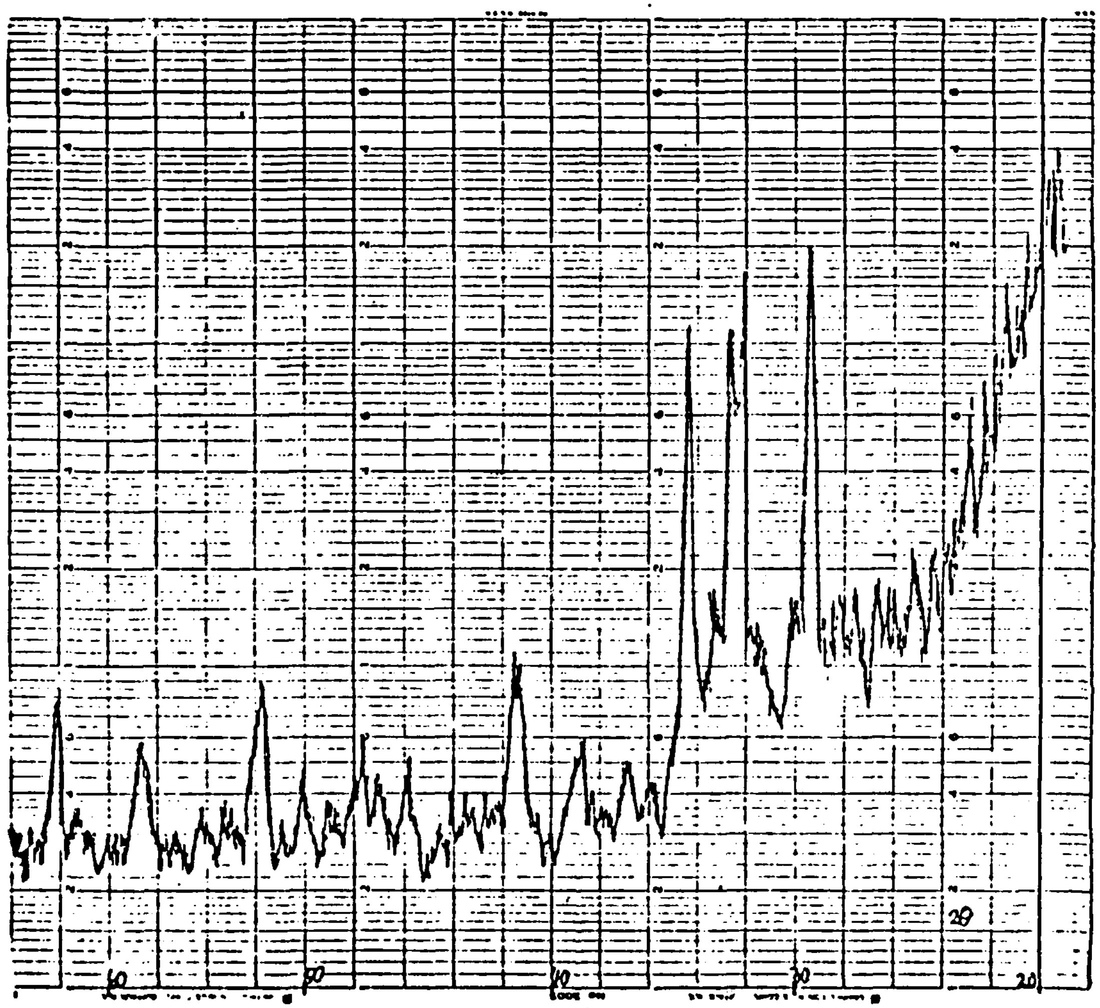


Fig. 63 - X-ray diffraction pattern of SET-45 cold weather paste with 1.75% borax mixed and cured at 200°F at 1 day.

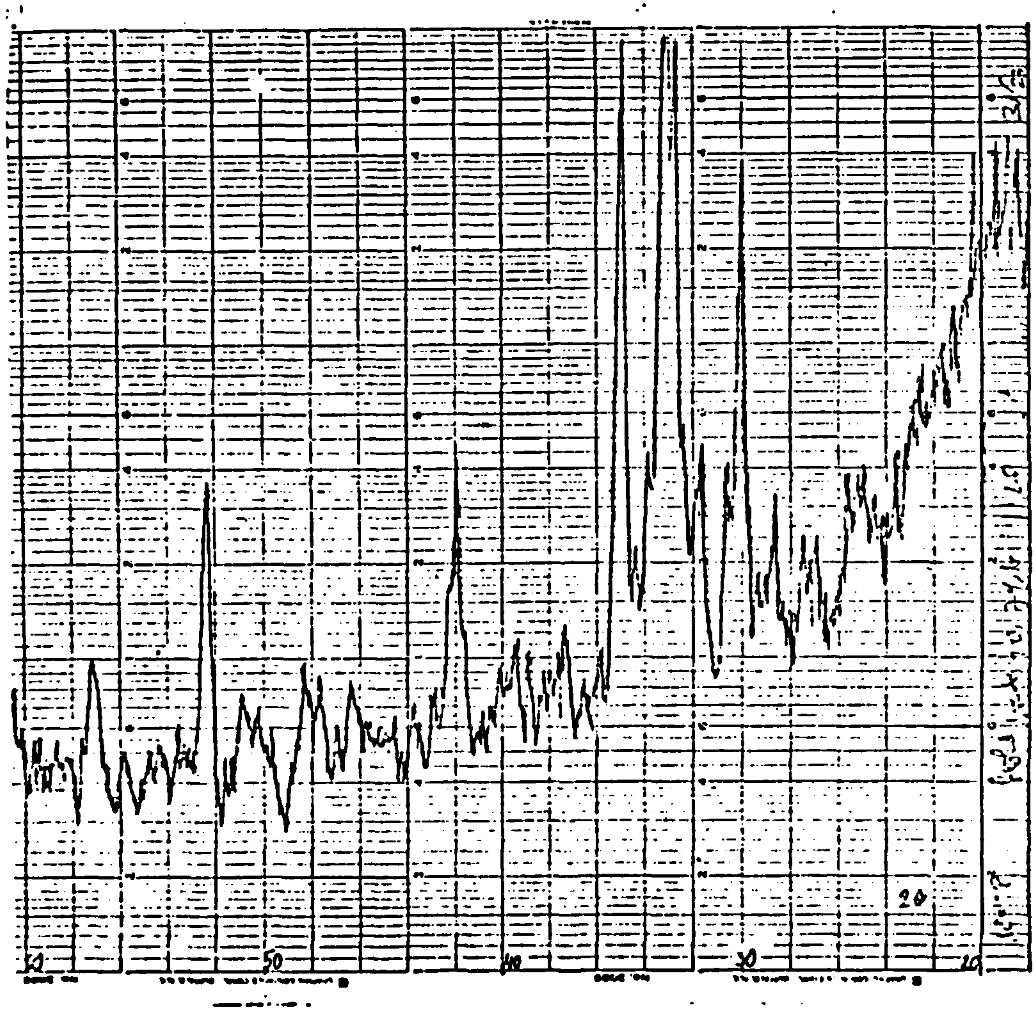


Fig. 64 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 200°F at 1 hour.

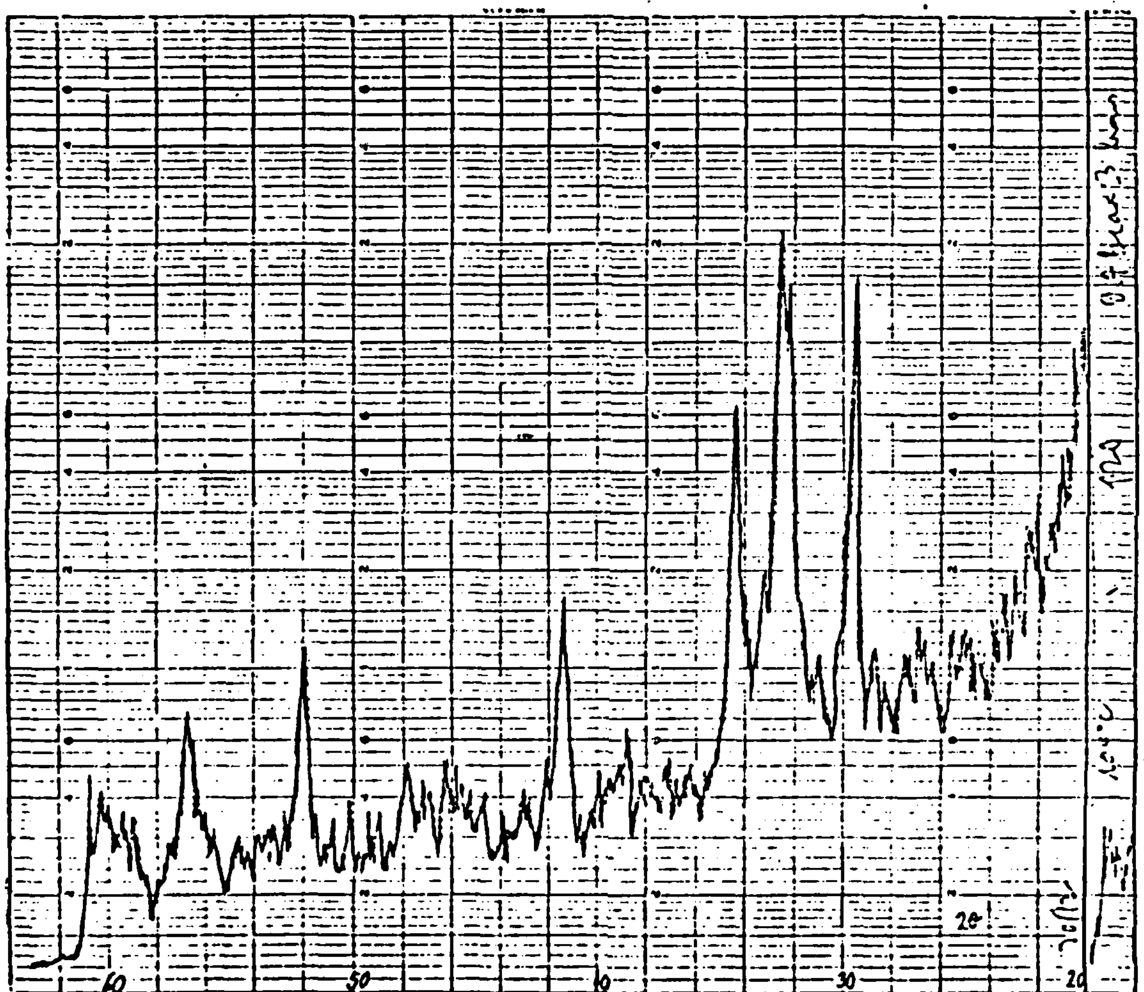


Fig. 65 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 200°F at [illegible] hours.

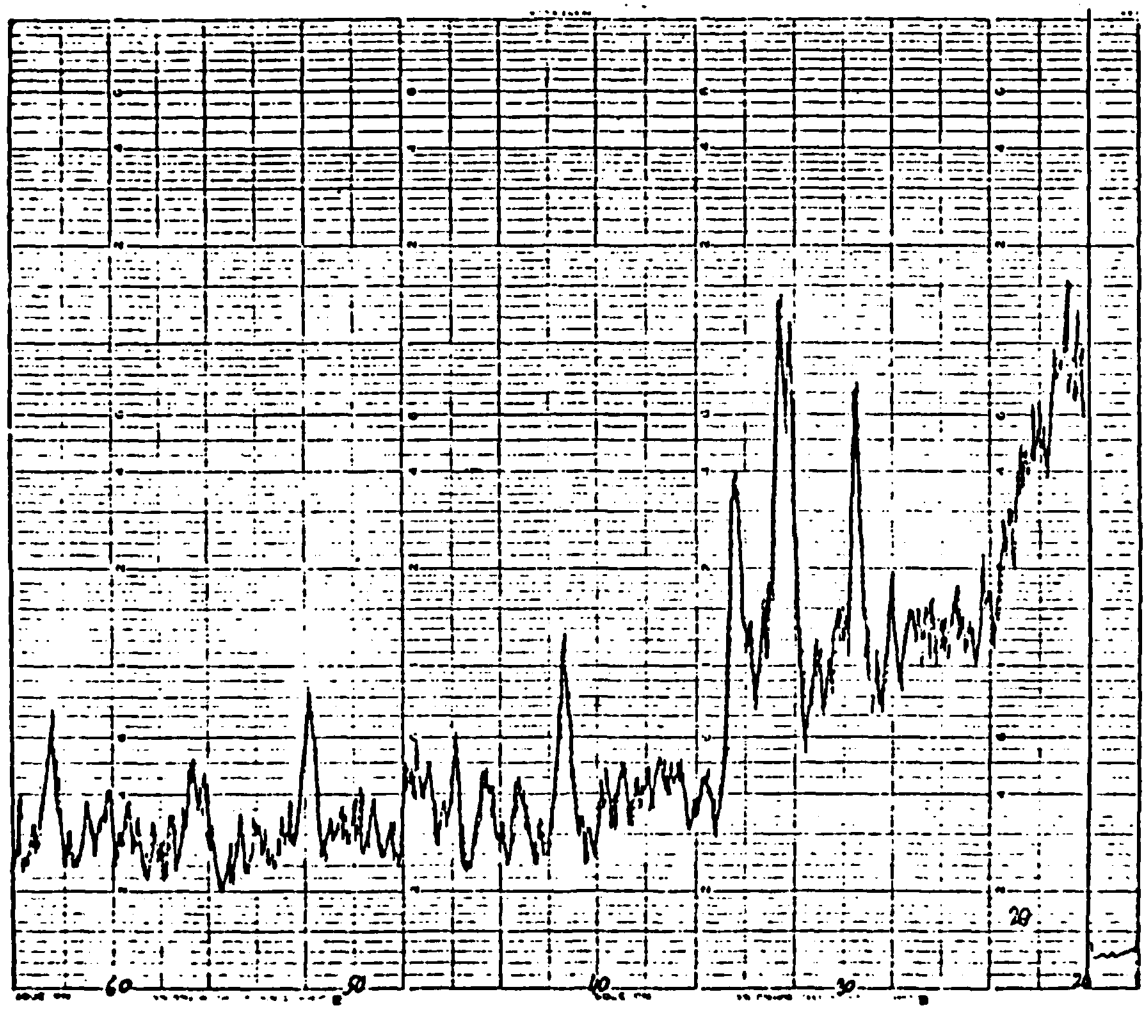


Fig. 66 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax
mixed and cured at 200°F at 1 day.

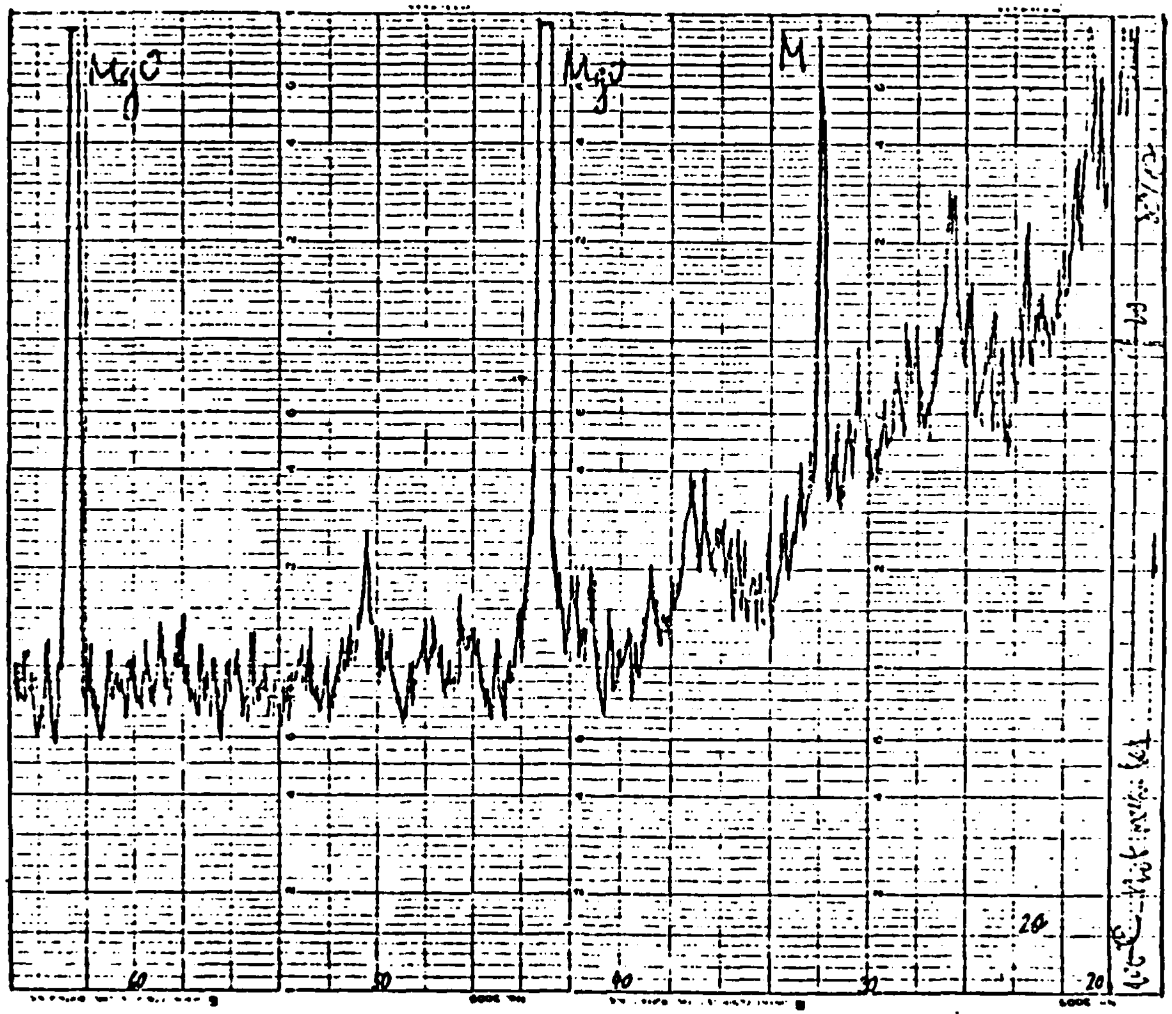


Fig. 67 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 200°F at 1 hr

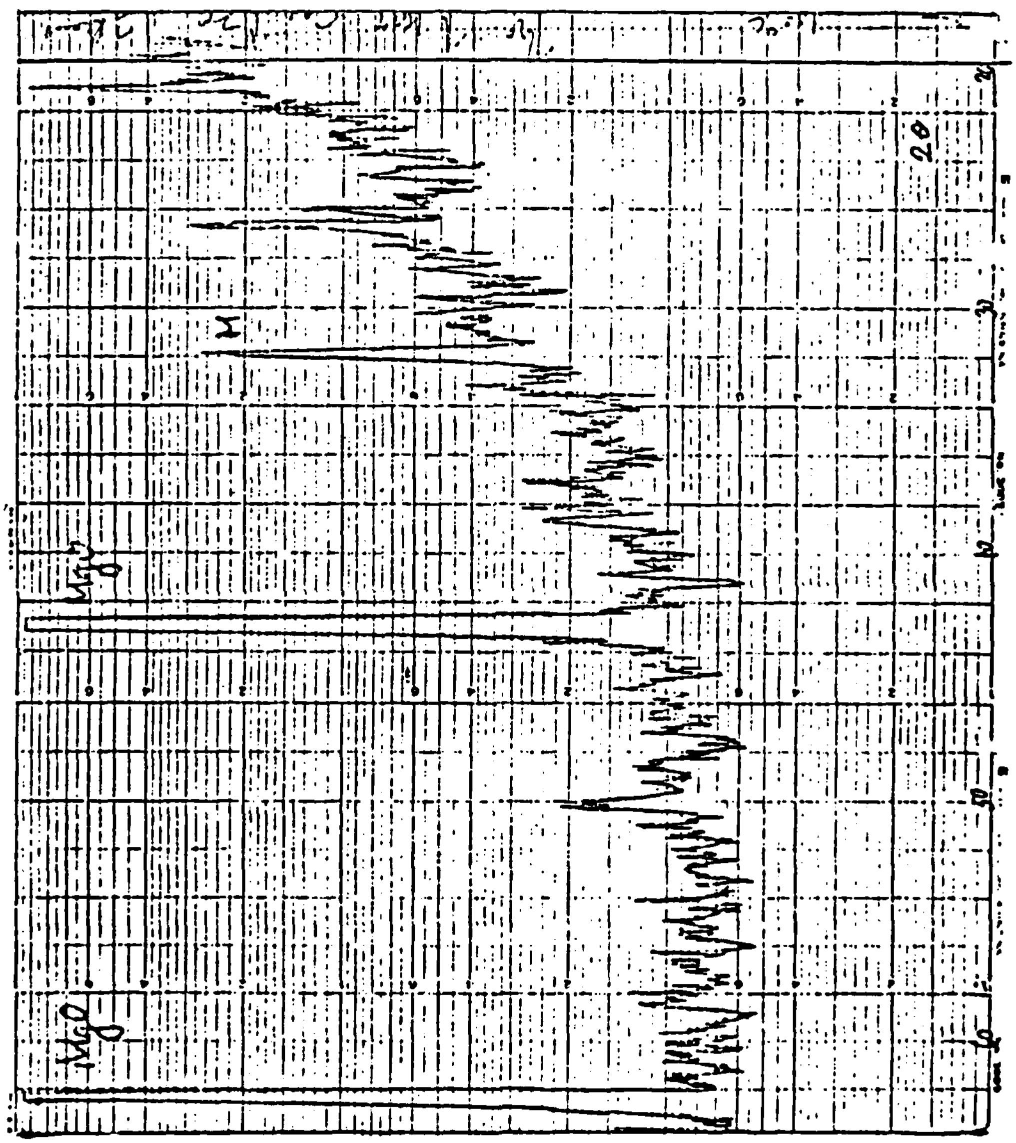


Fig. 68 - X-ray diffraction pattern of SET-45 hot paste mixed and cured at 200°F at 3 hours.

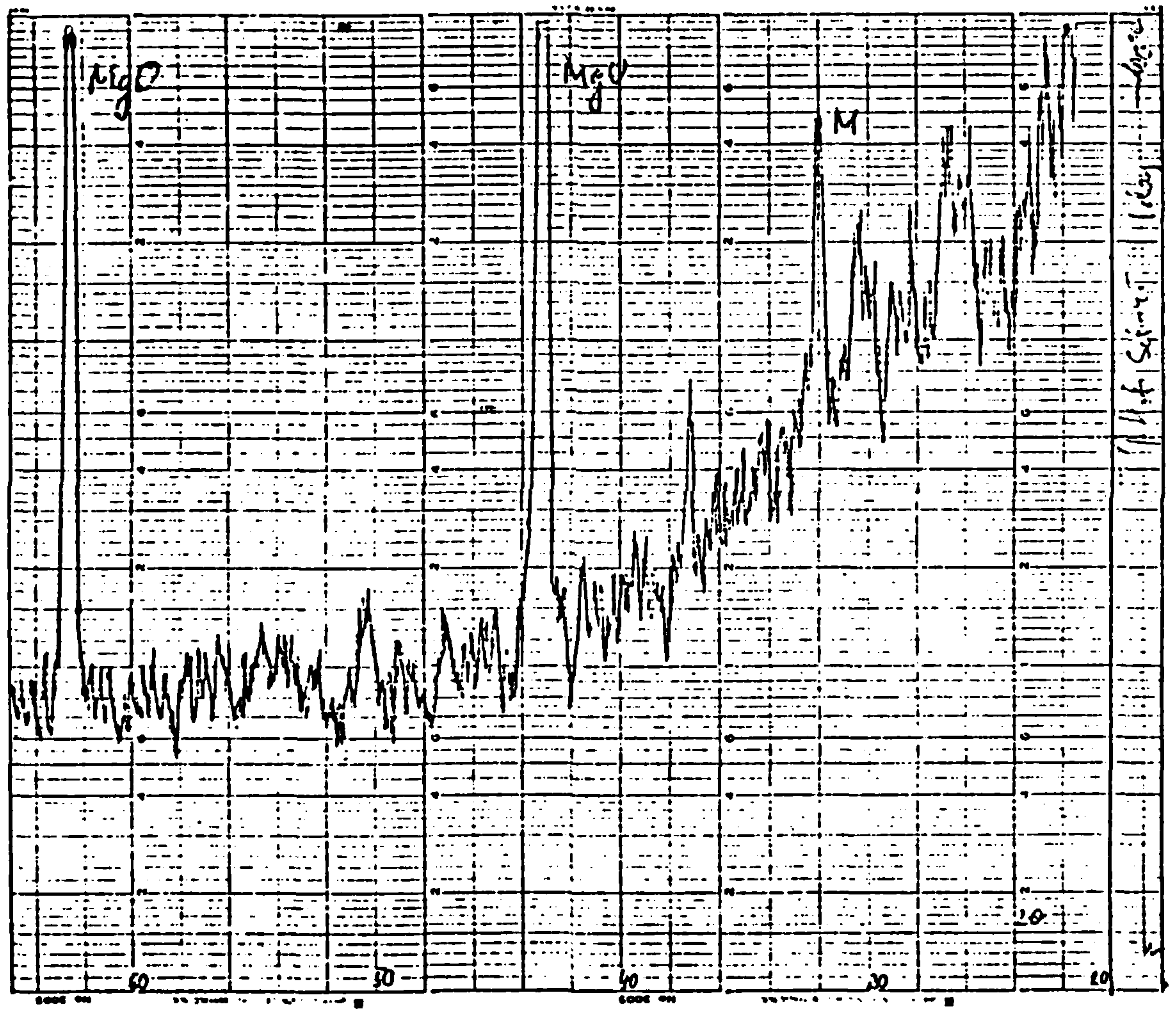


Fig. 69 - X-ray diffraction pattern of SET-45 hot paste mixed and cured at 200°F at 1 day.

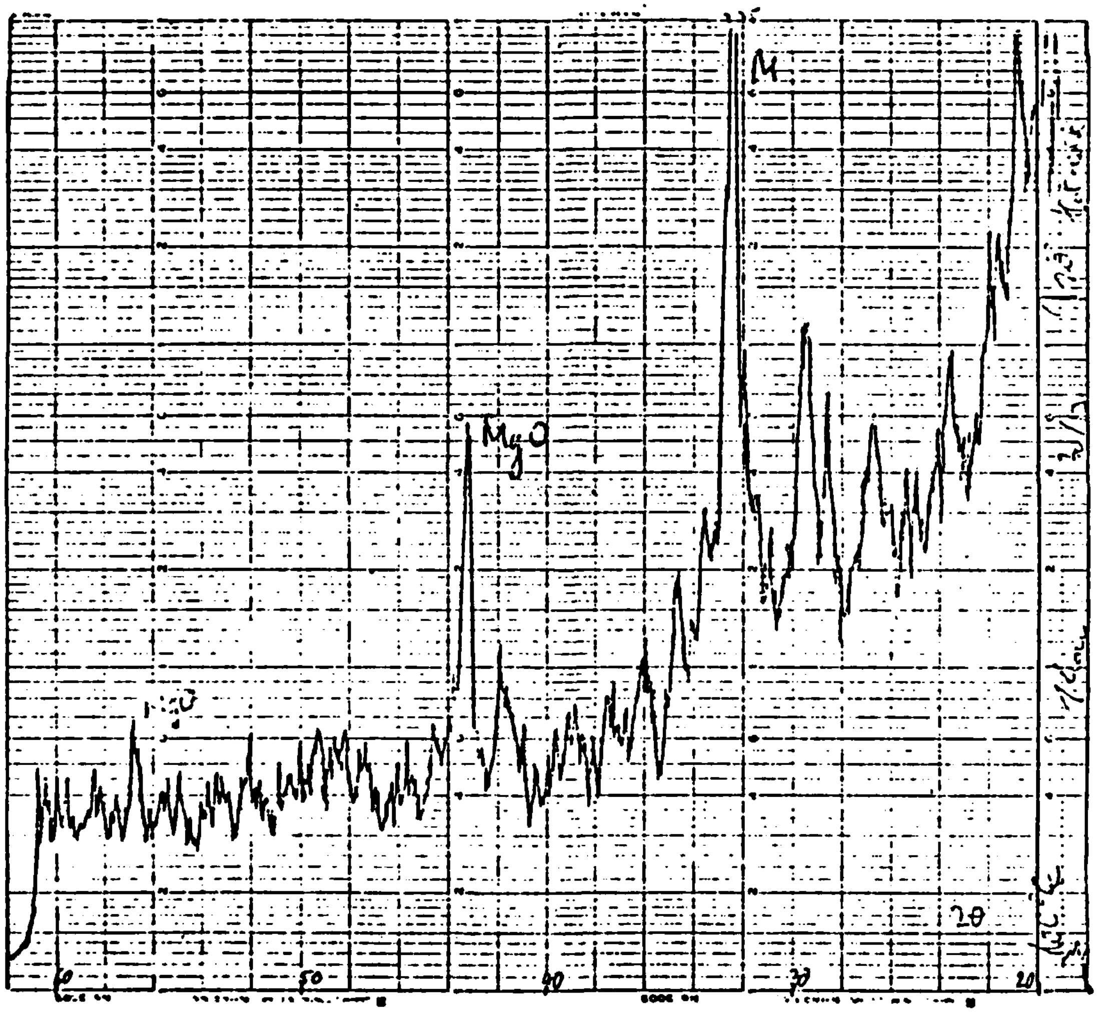


Fig. 70 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 200°F at 1 hour.

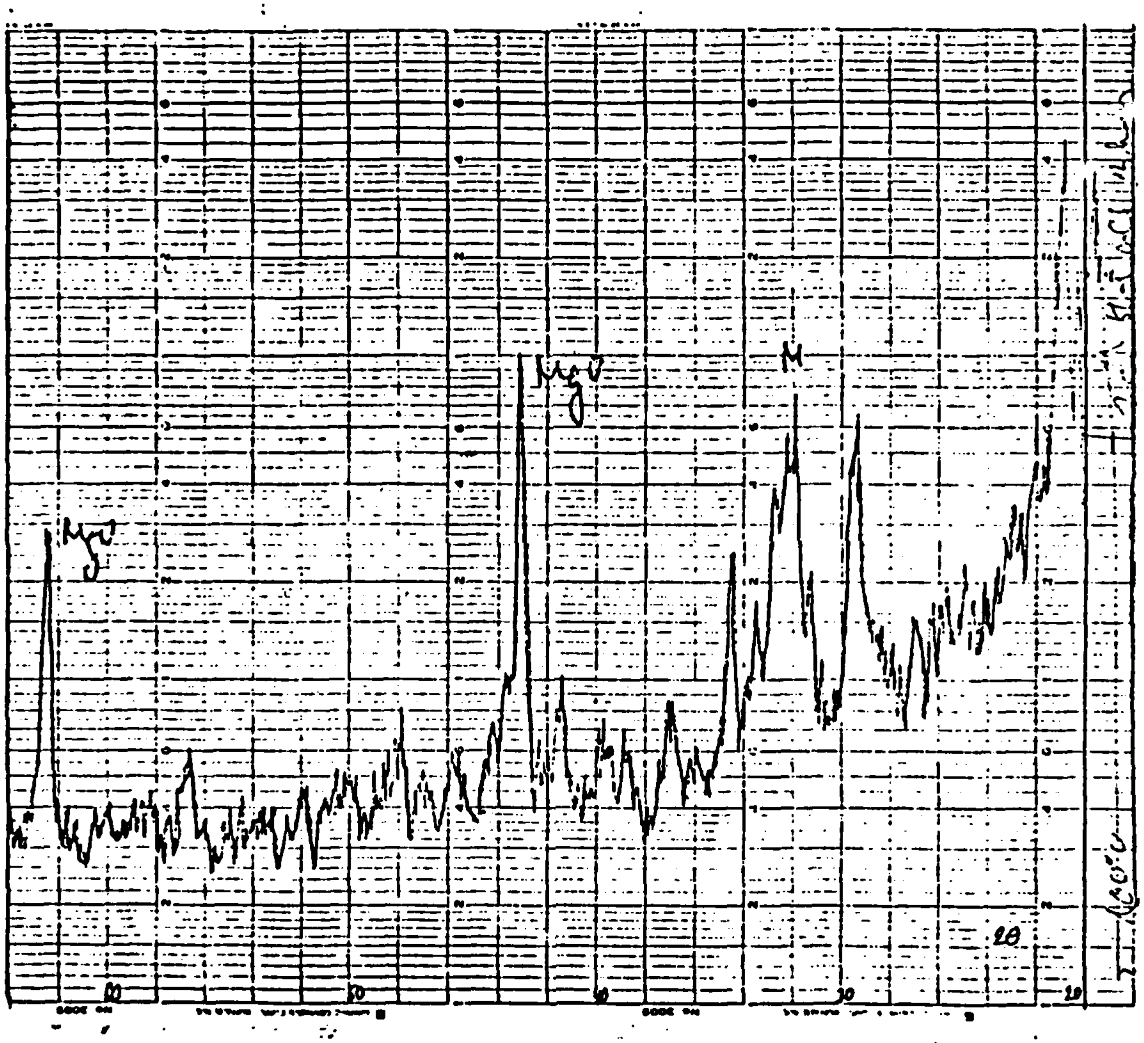


Fig. 71 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 200°F at 3 hours.

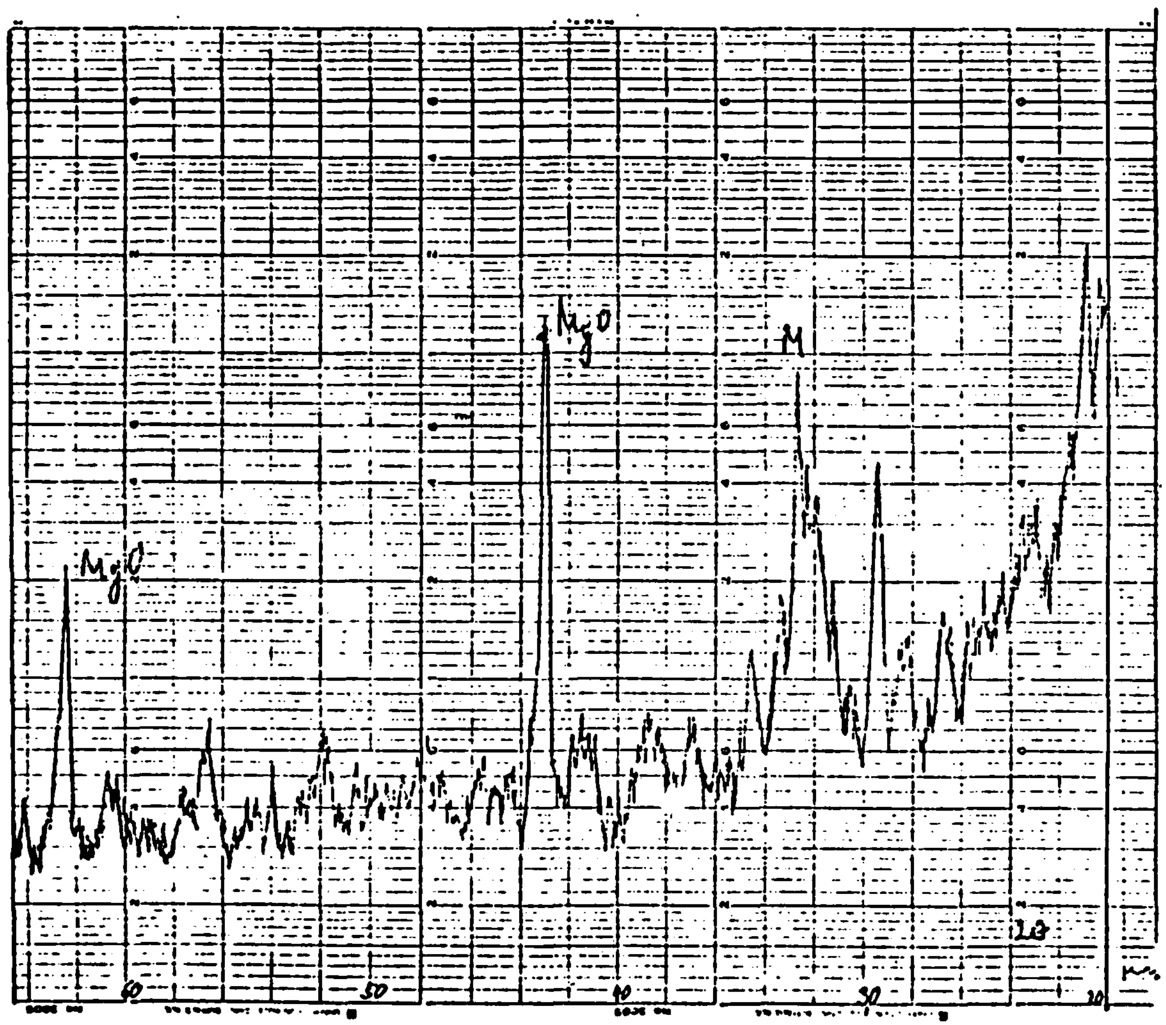


Fig. 72 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 200°F at 1 day.

Fig. 73 - SEM picture of SET-45 cold weather paste dry-cured at 1 month. M = 3000X.

Fig. 74 - SEM picture of SET-45 cold weather paste with polyphosphate dry-cured at 1 month. M = 3000X.

Fig. 75 - SEM picture of SET-45 cold weather paste wet-cured at 1 month. M = 3000X

Fig. 76 - SEM picture of SET-45 cold weather paste with polyphosphate wet-cured at 1 month. M = 3000X.

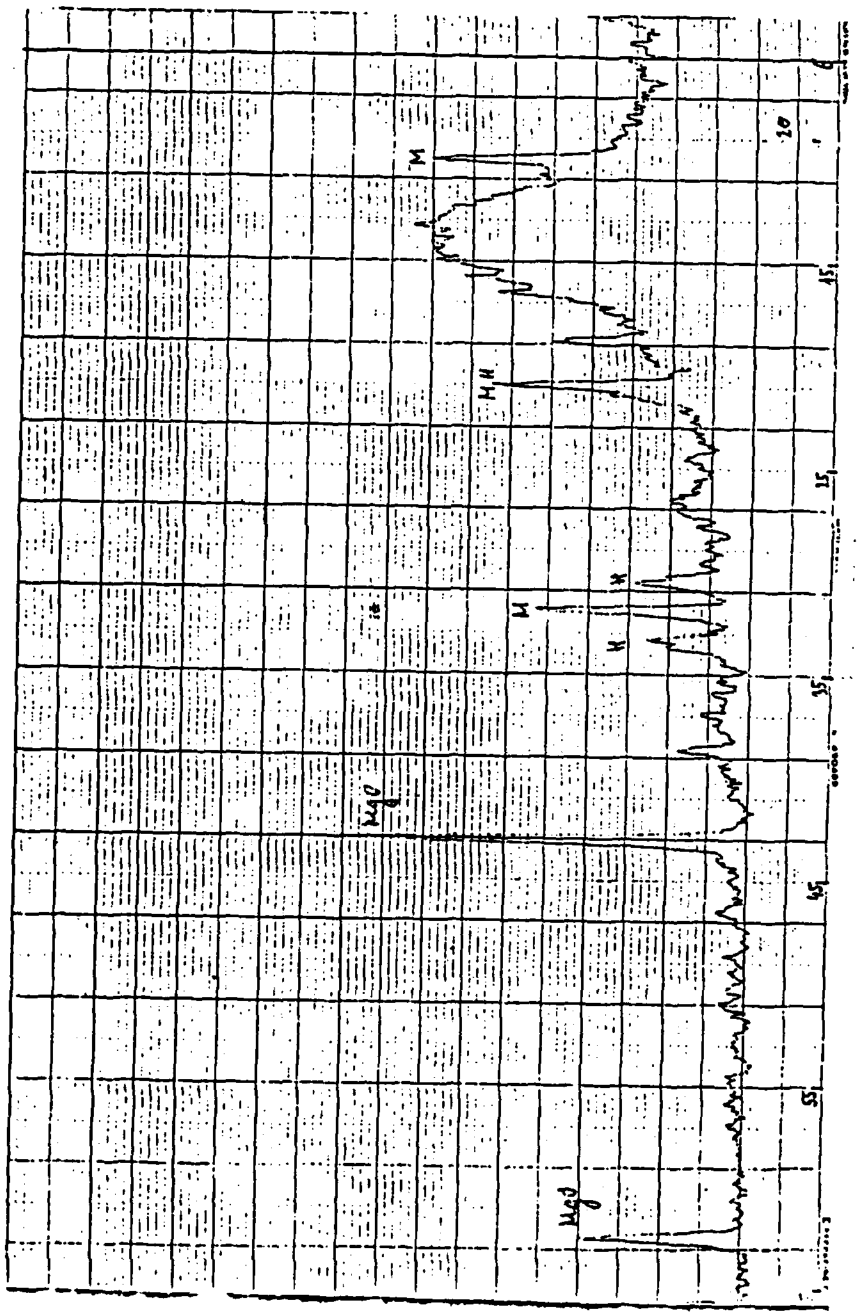


Fig. 77 - X-ray diffraction pattern of SET-45 cold weather paste dry-cured at 1 month.

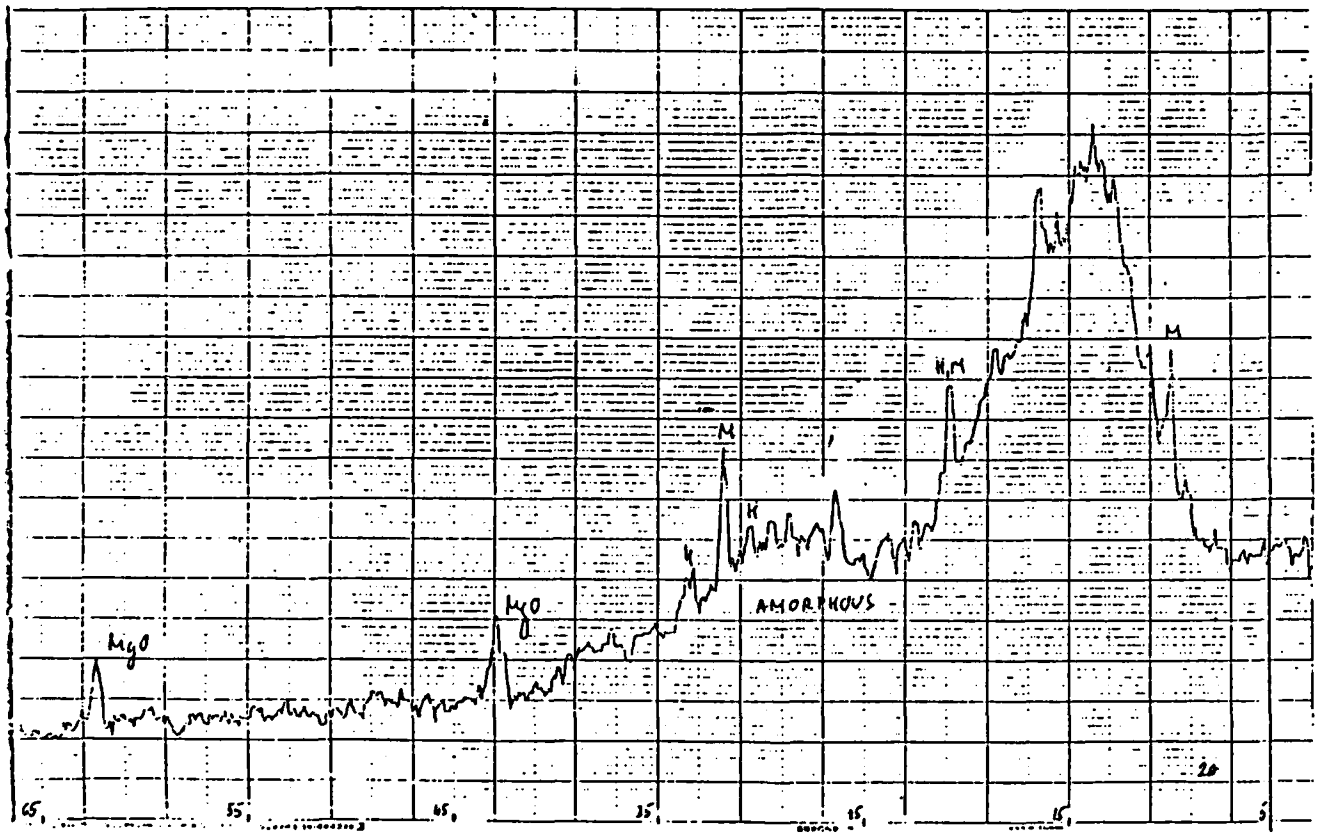


Fig. 78 - X-ray diffraction pattern of SET-45 cold weather paste wet-cured at 1 month.

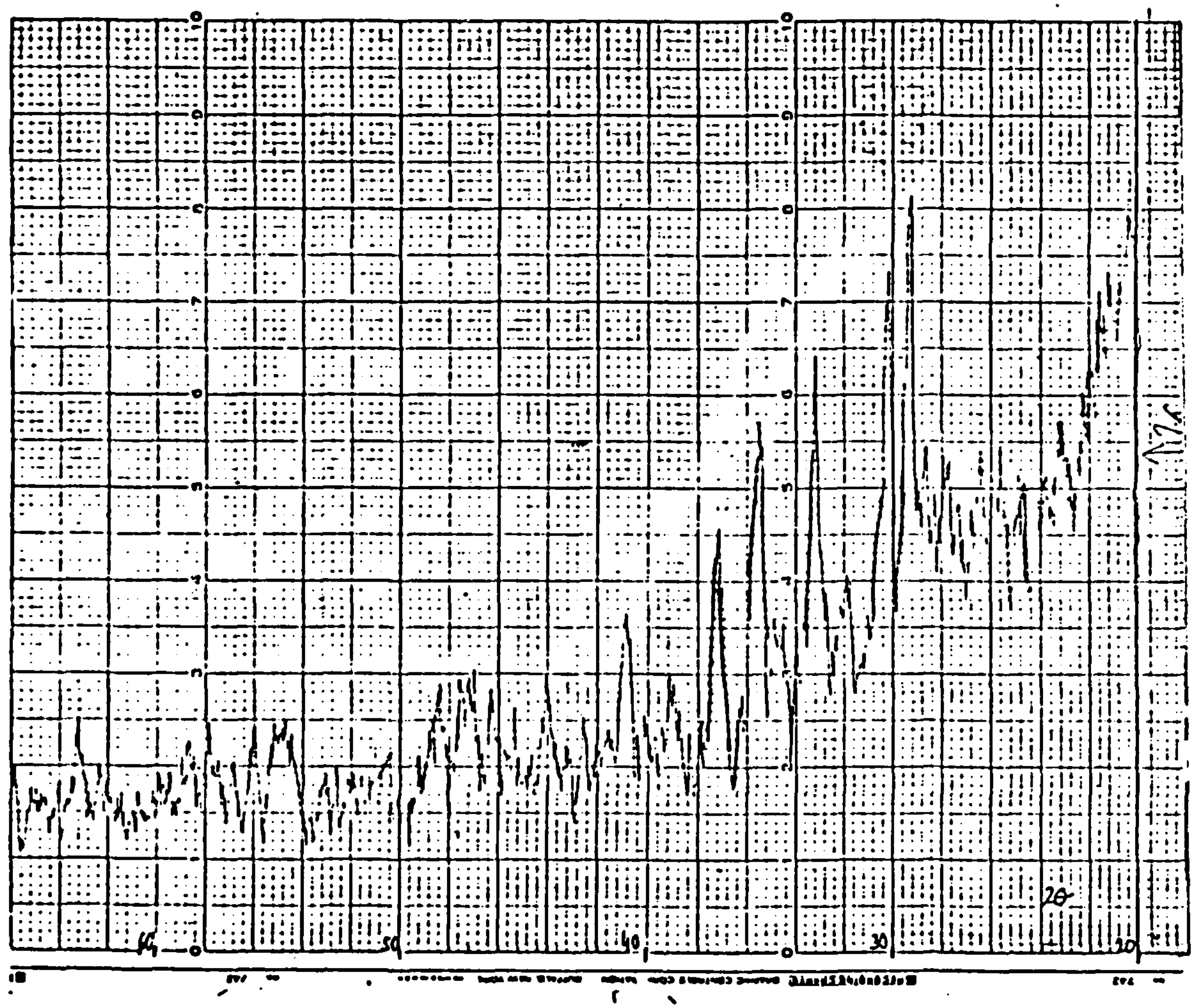


Fig. 79 - X-ray diffraction pattern of the thin layer on the surface of 3 months old wet-cured SET-45 cold weather paste.

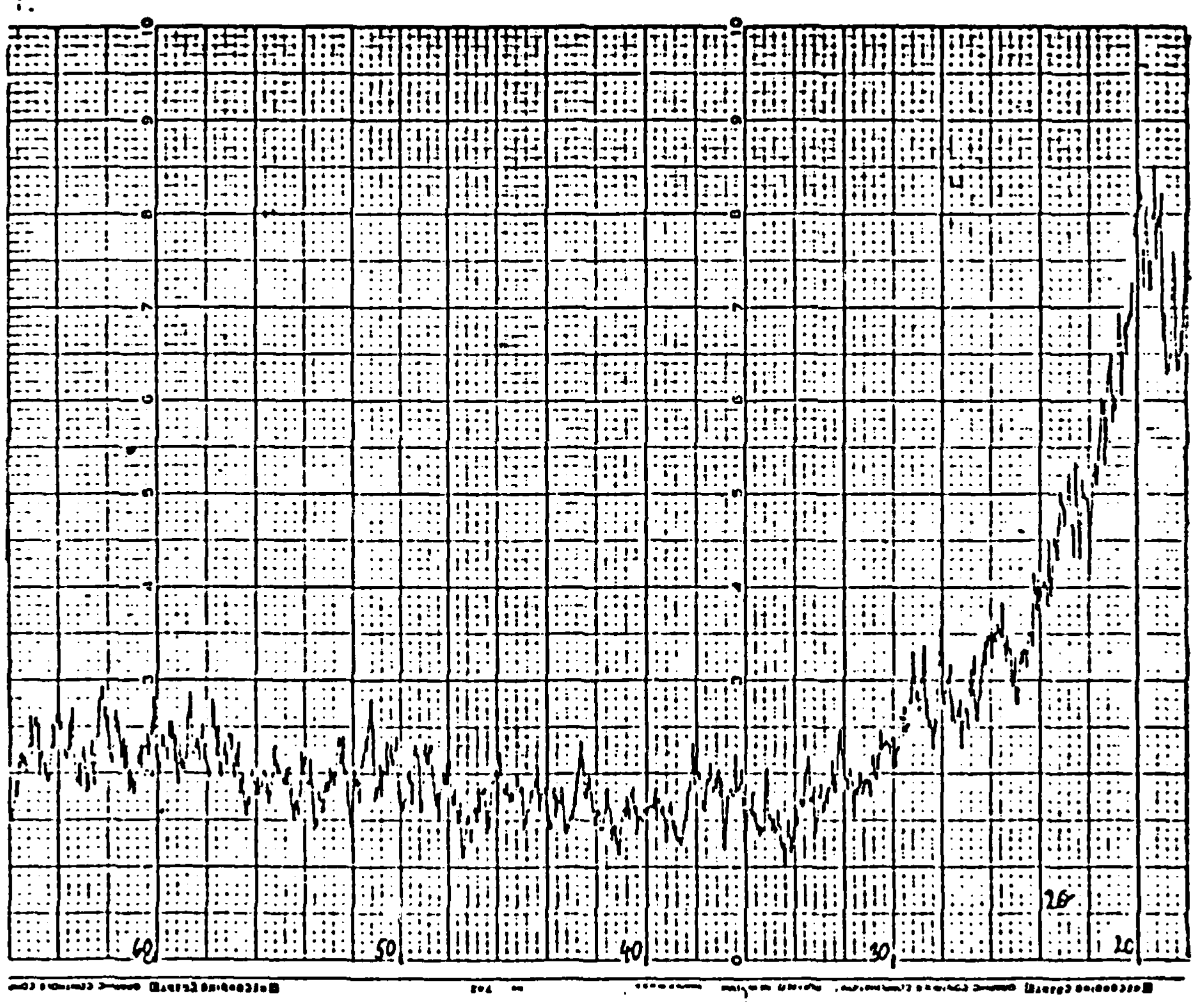


Fig. [illegible] pattern [illegible] months old wet-cured SET-45 cold weather paste.

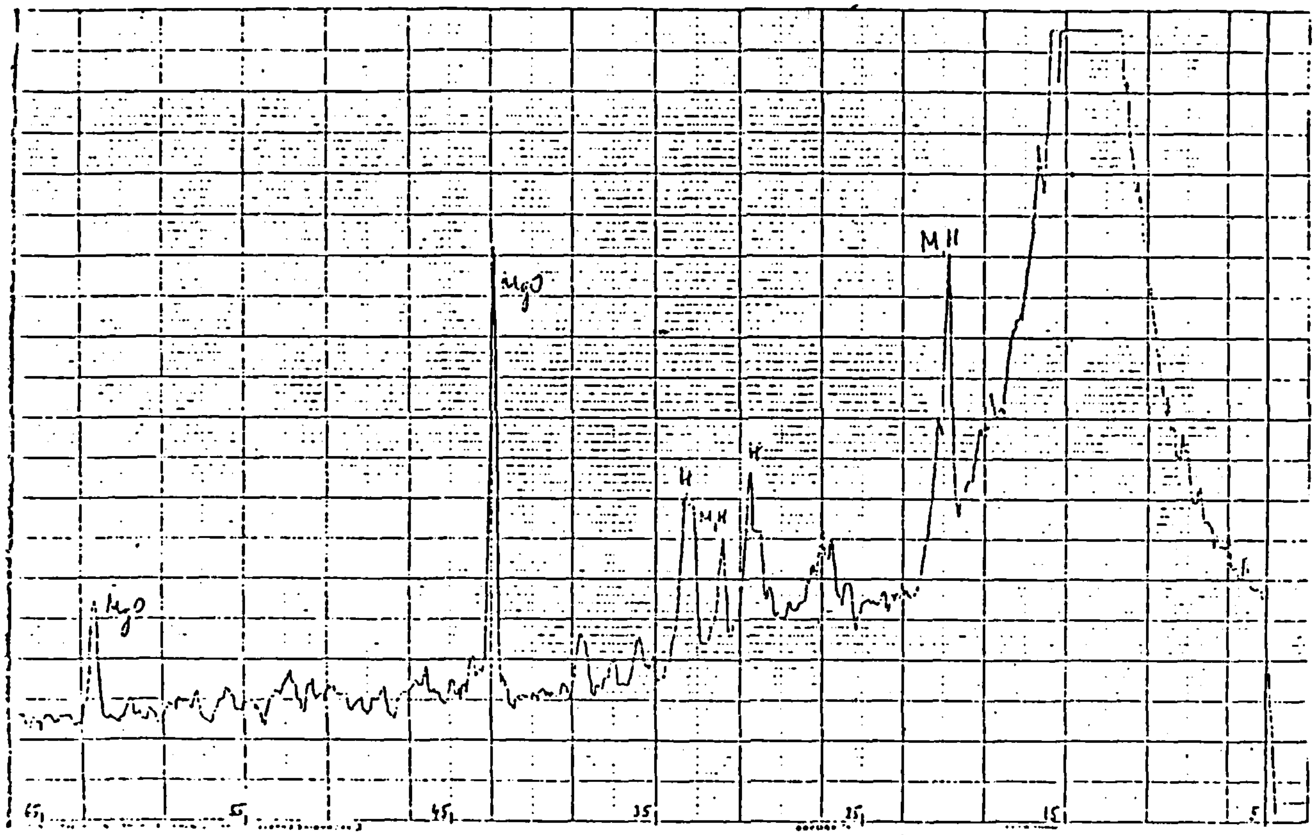


Fig. 81 - X-ray diffraction pattern of SET-45 cold weather paste with polyphosphate dry-cured at 1 month.

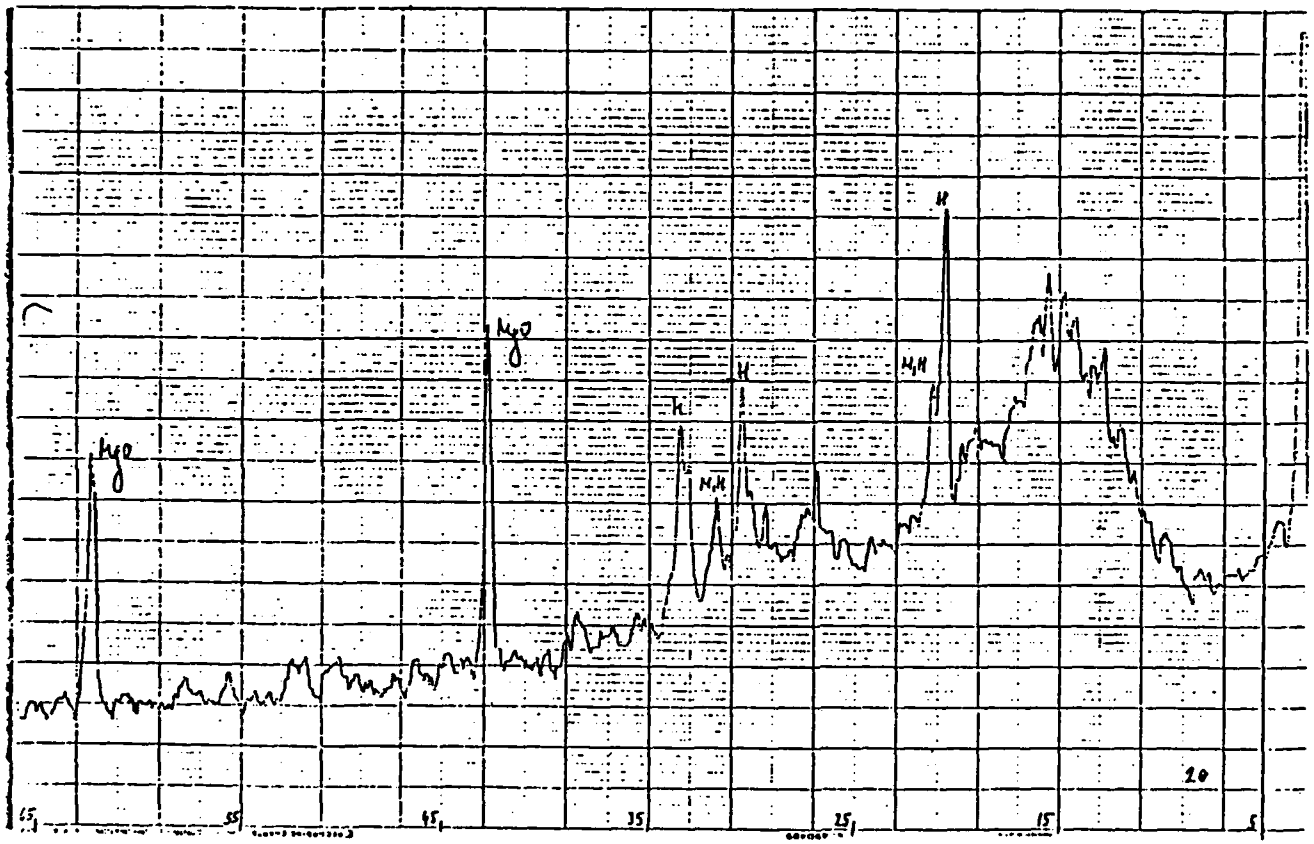


Fig. 82 - X-ray diffraction pattern of SET-45 cold weather paste with polyphosphate wet-cured at 1 month.

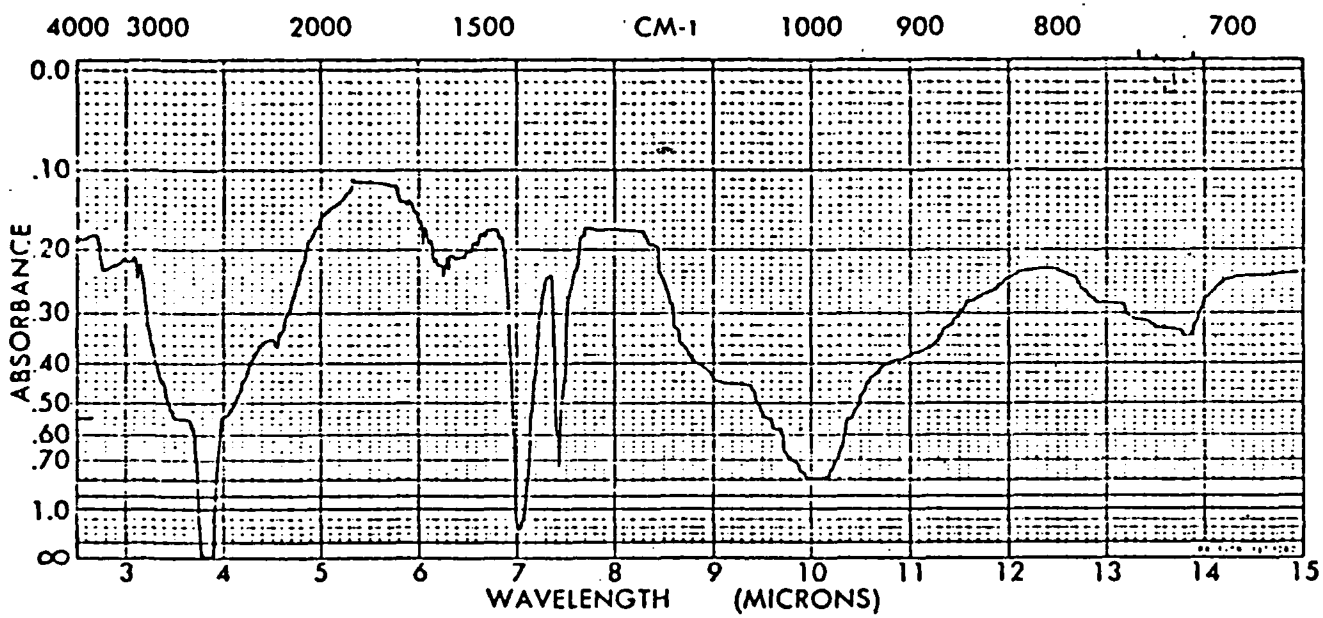


Fig. 83 - IR spectrum of SET-45 cold weather paste with polyphosphate dry-cured at 1 month.

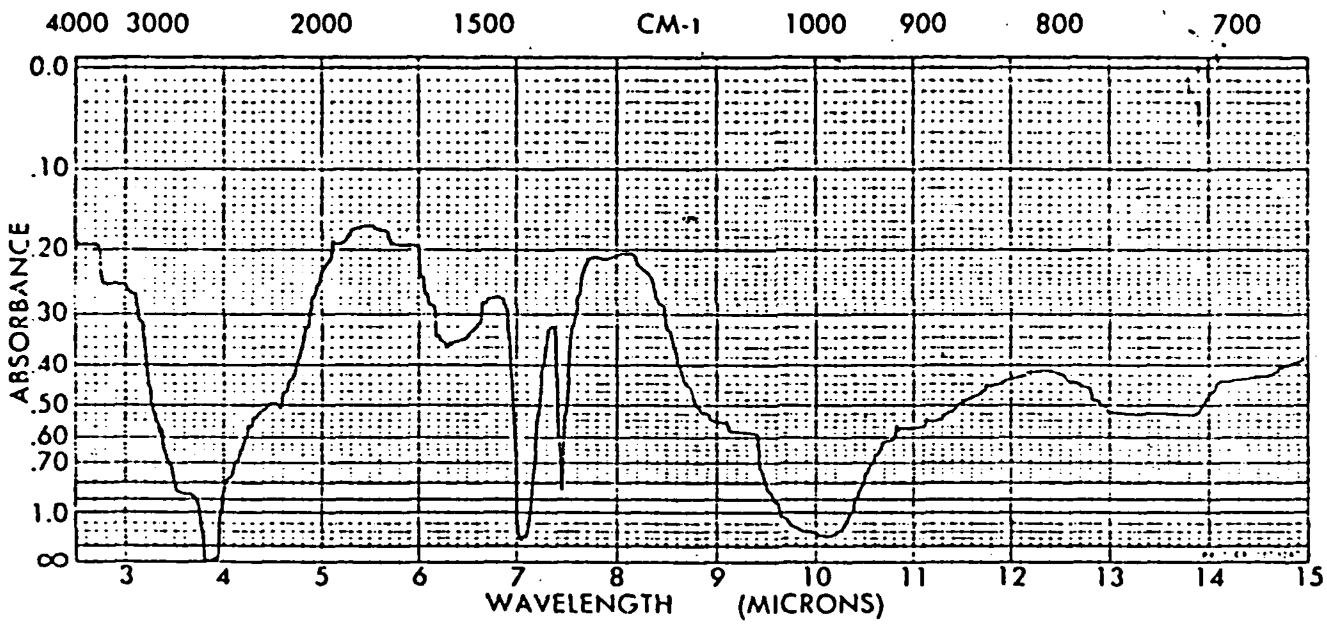


Fig. 84 - IR spectrum of SET-45 cold weather paste with polyphosphate wet-cured at 1 month.

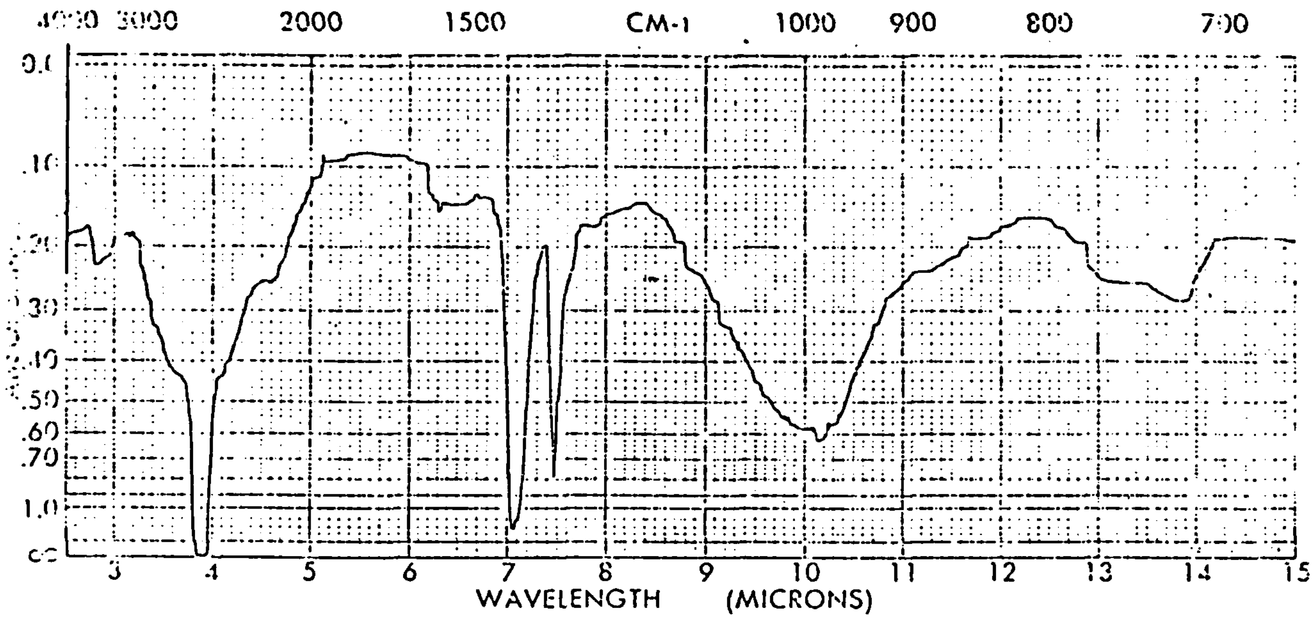


Fig. 85 - IR Spectrum of SET-45 cold weather paste dry-cured at 1 month.

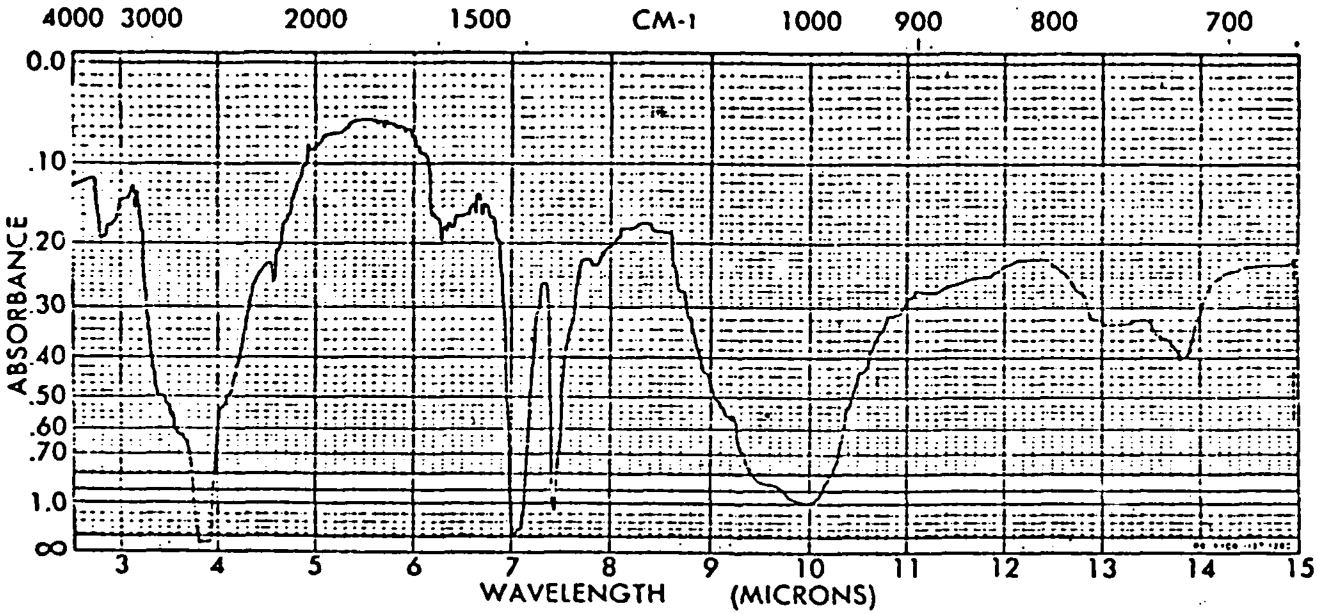


Fig. 86 - IR spectrum of SET-45 cold weather paste wet-cured at 1 month.

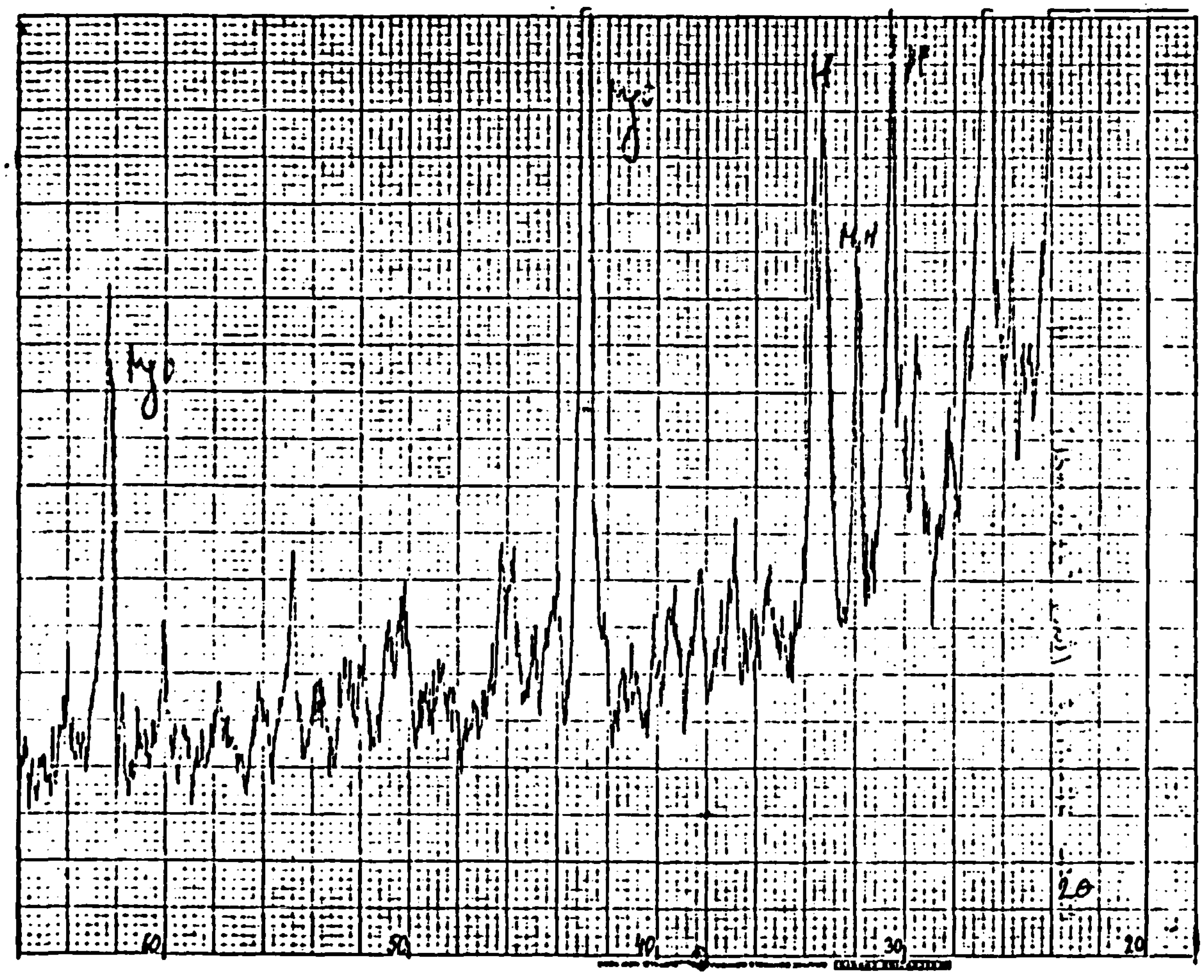


Fig. 87 - X-ray diffraction pattern of SET-45 cold weather mortar before curing at 1 hour.

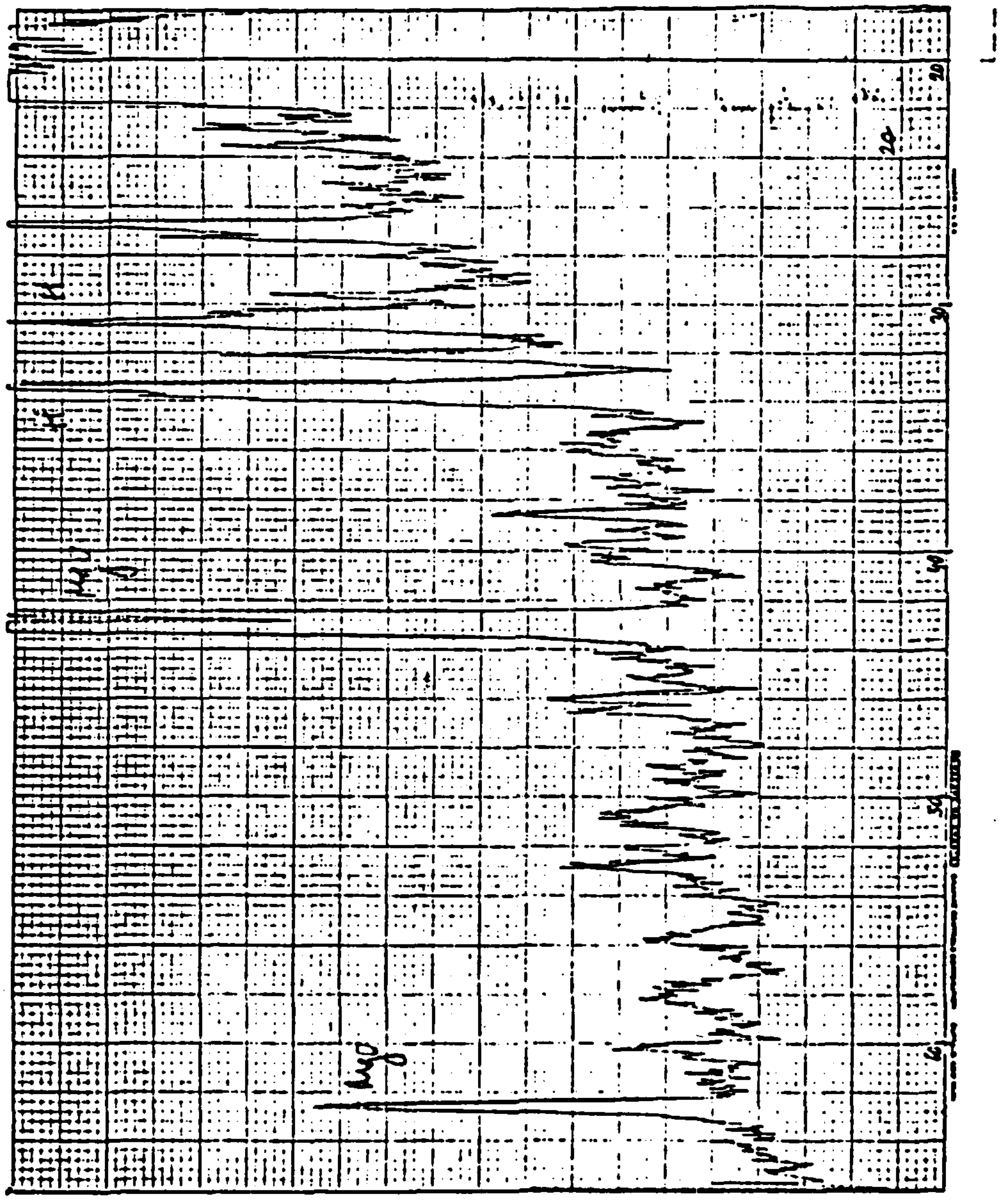


Fig. 88 - X-ray diffraction of SET-45 cold weather mortar dry-cured at 3 hours.

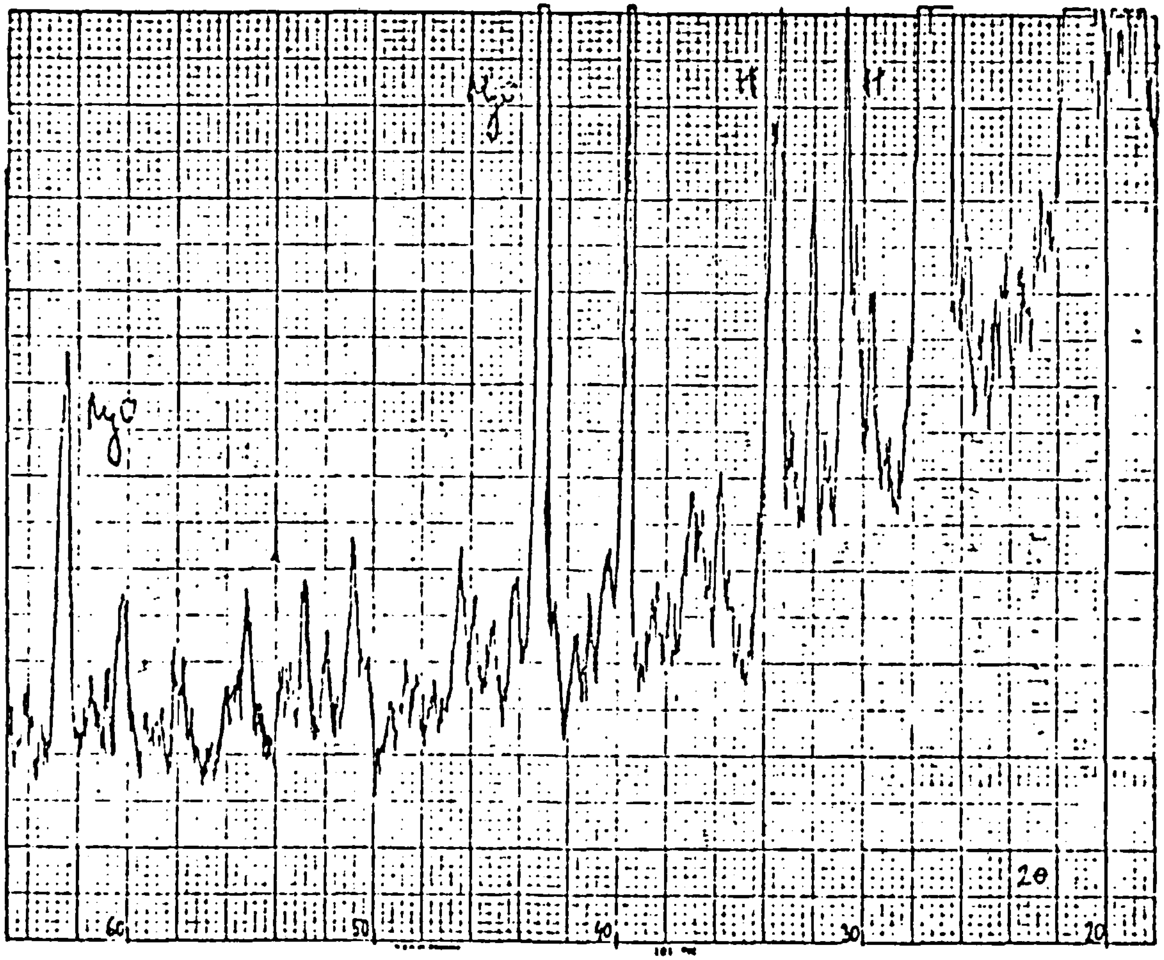


Fig. 89 - X-ray diffraction pattern of SET-45 cold weather mortar wet-cured at 3 hours.

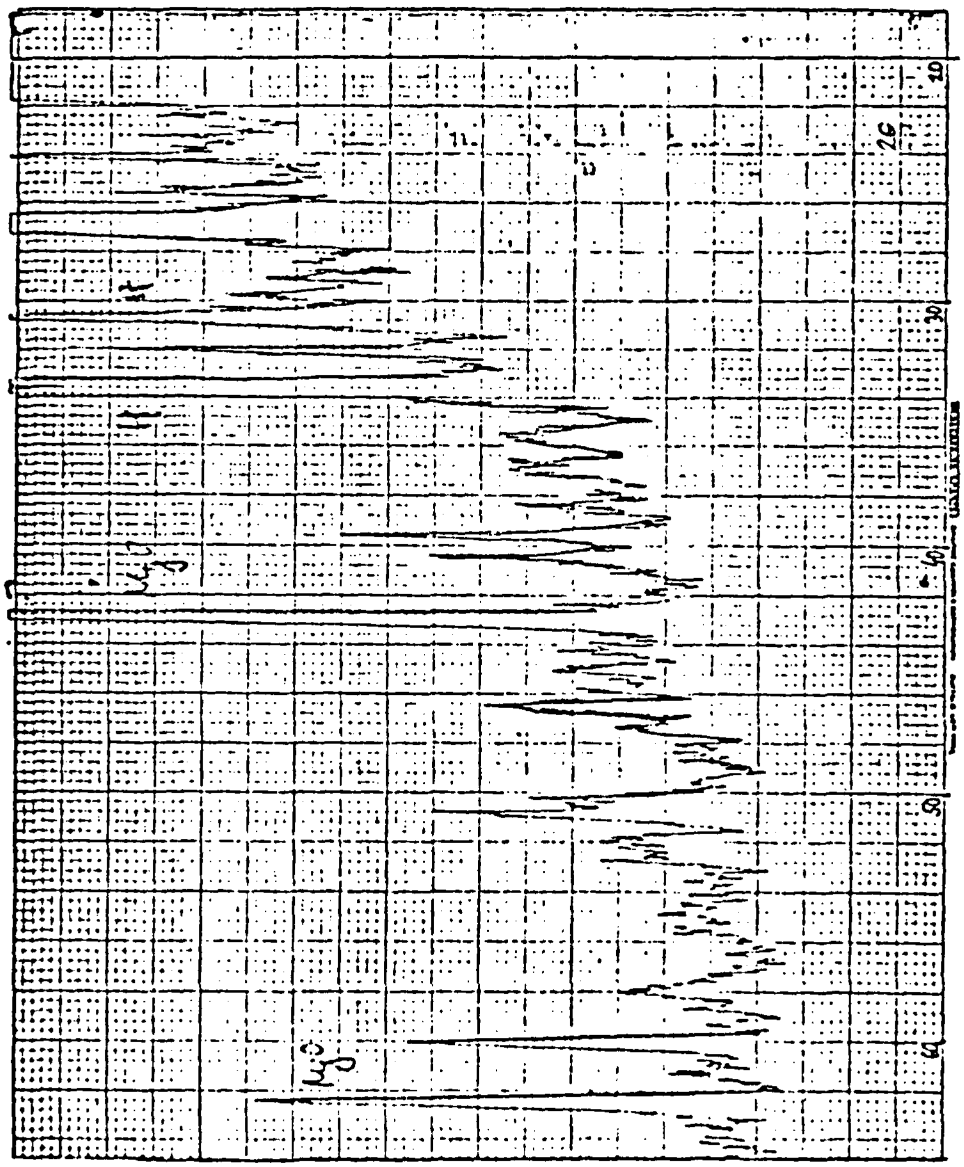


Fig. 90 - X-ray diffraction pattern of SET-45 cold weather mortar dry-cured at 1 day.

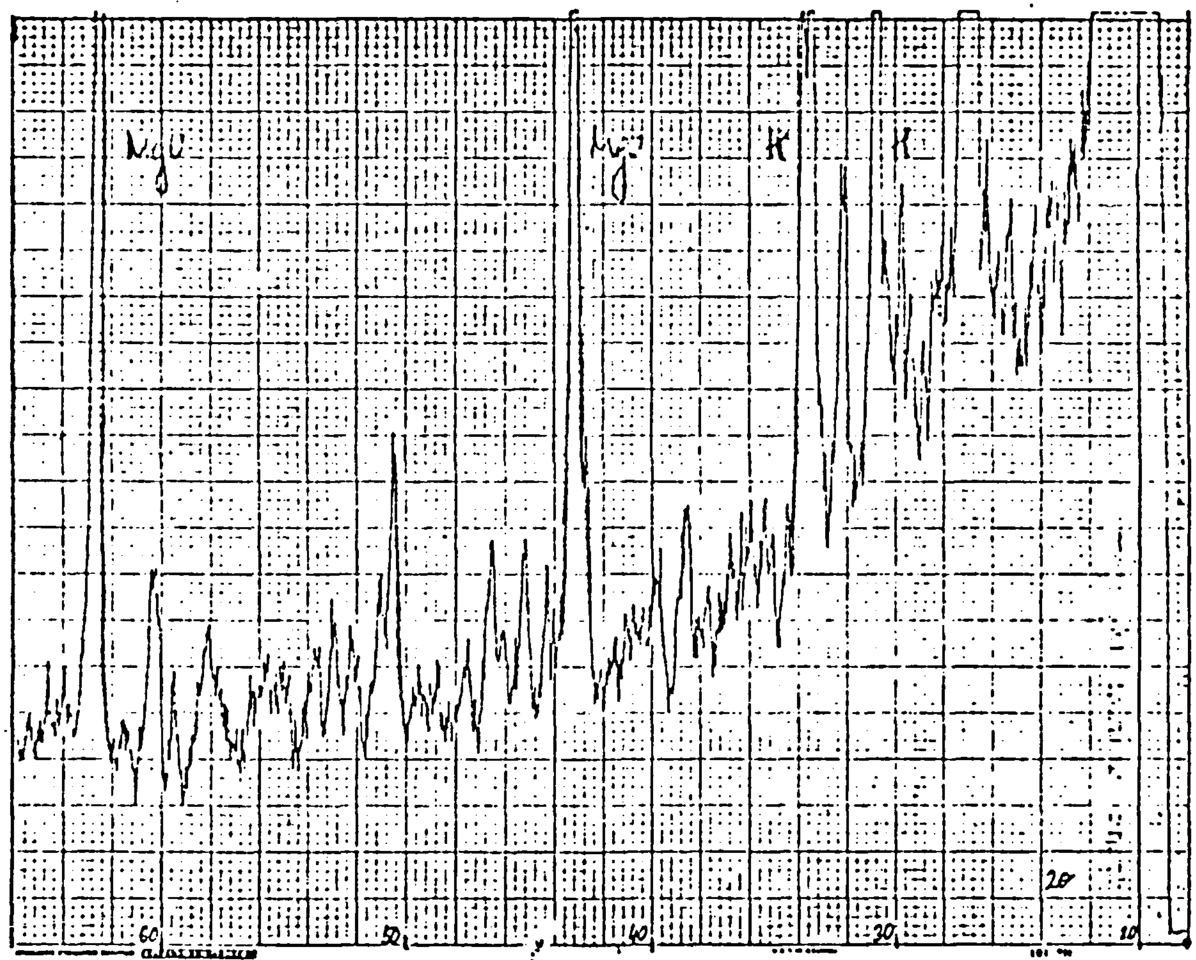


Fig. 91 - X-ray diffraction pattern of SET-45 cold weather mortar wet-cured at 1 day.

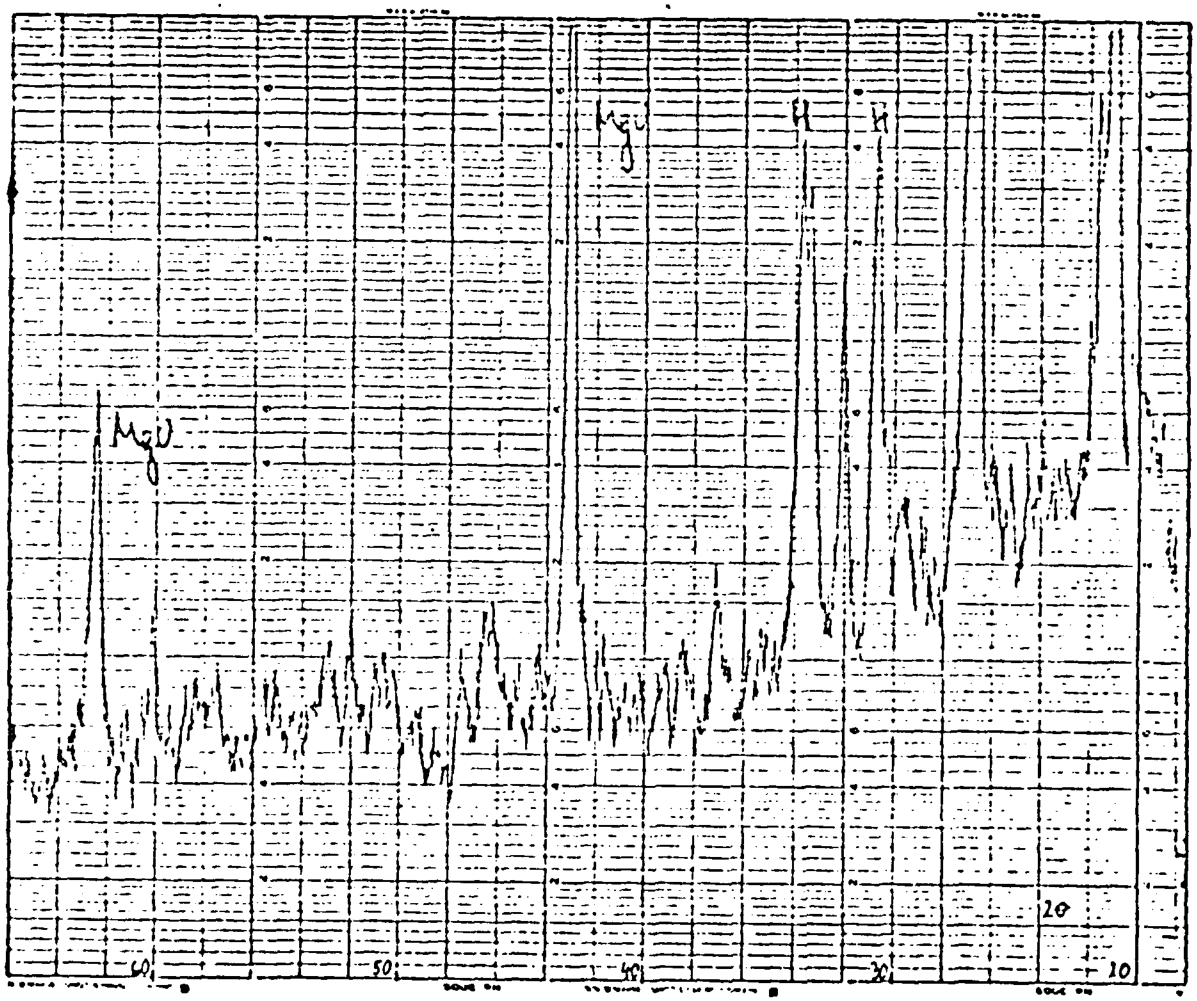


Fig. 92 - X-ray diffraction pattern of SET-45 cold weather mortar dry-cured at 1 week.

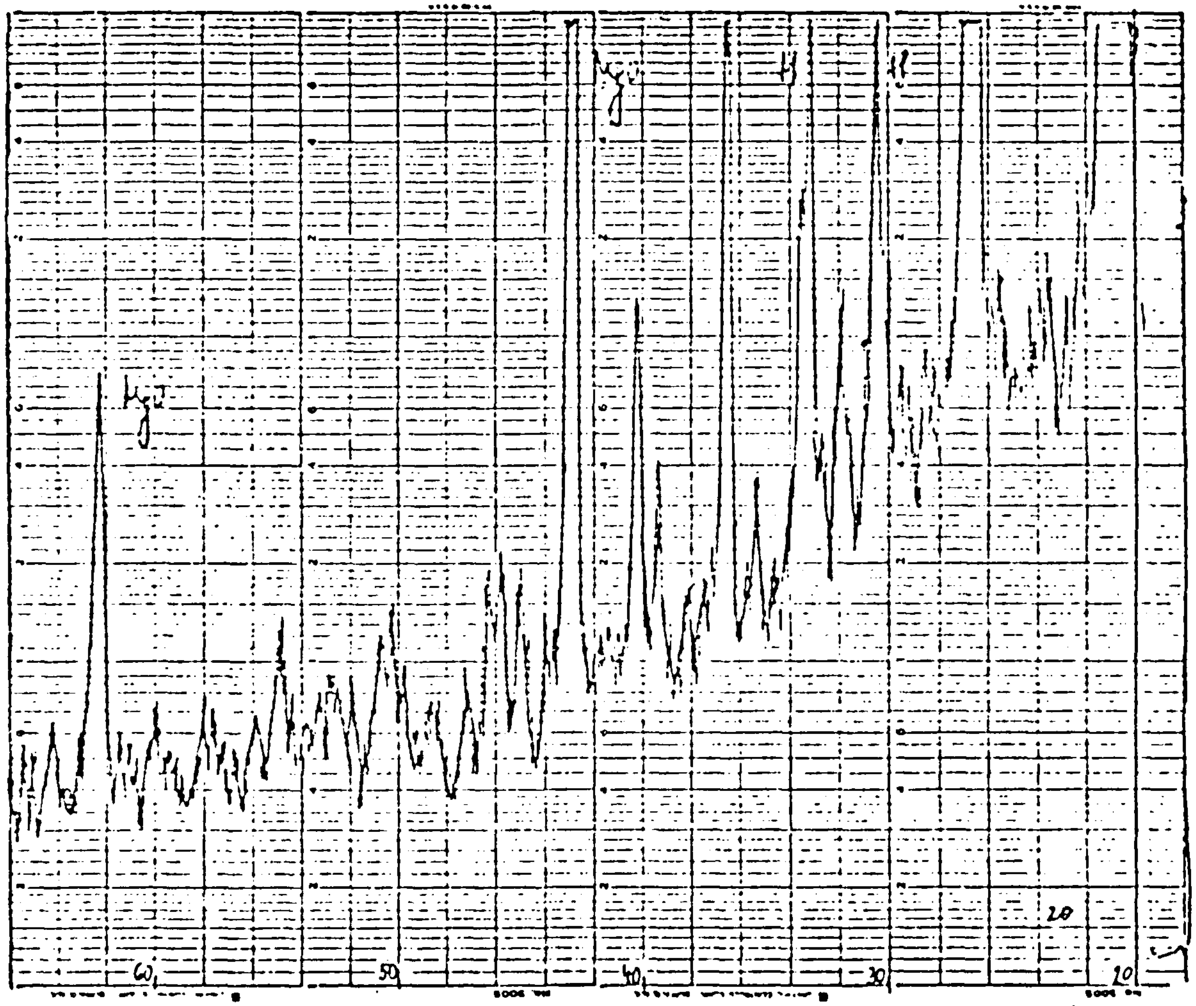


Fig. 93 - X-ray diffraction pattern of SET-45 cold weather mortar wet-cured at 1 week.

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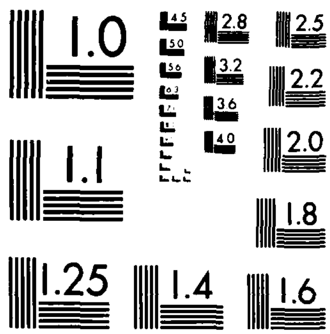

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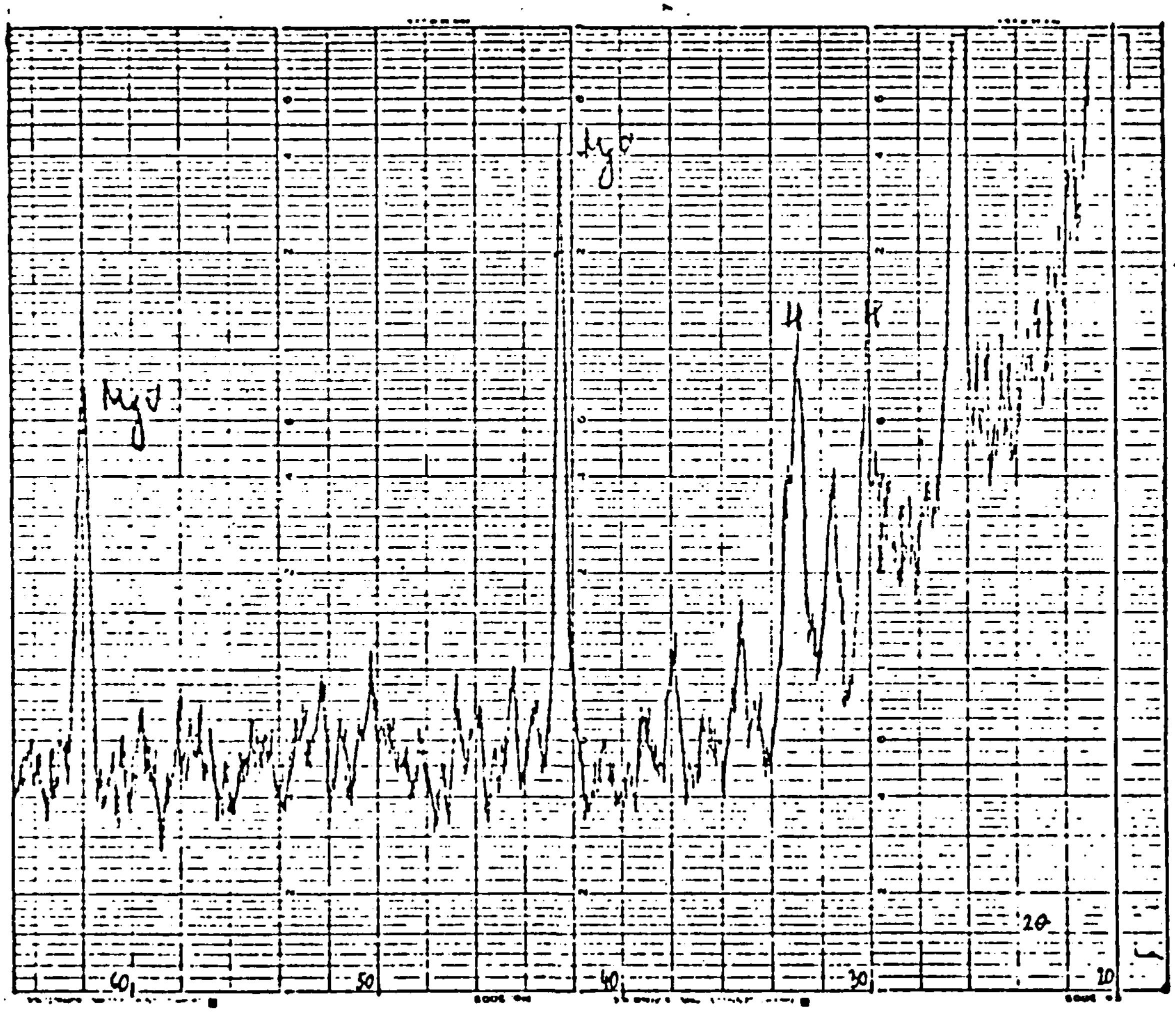

Fig. 94 - X-ray diffraction pattern of SET-45 cold weather mortar dry-cured at 1 month.

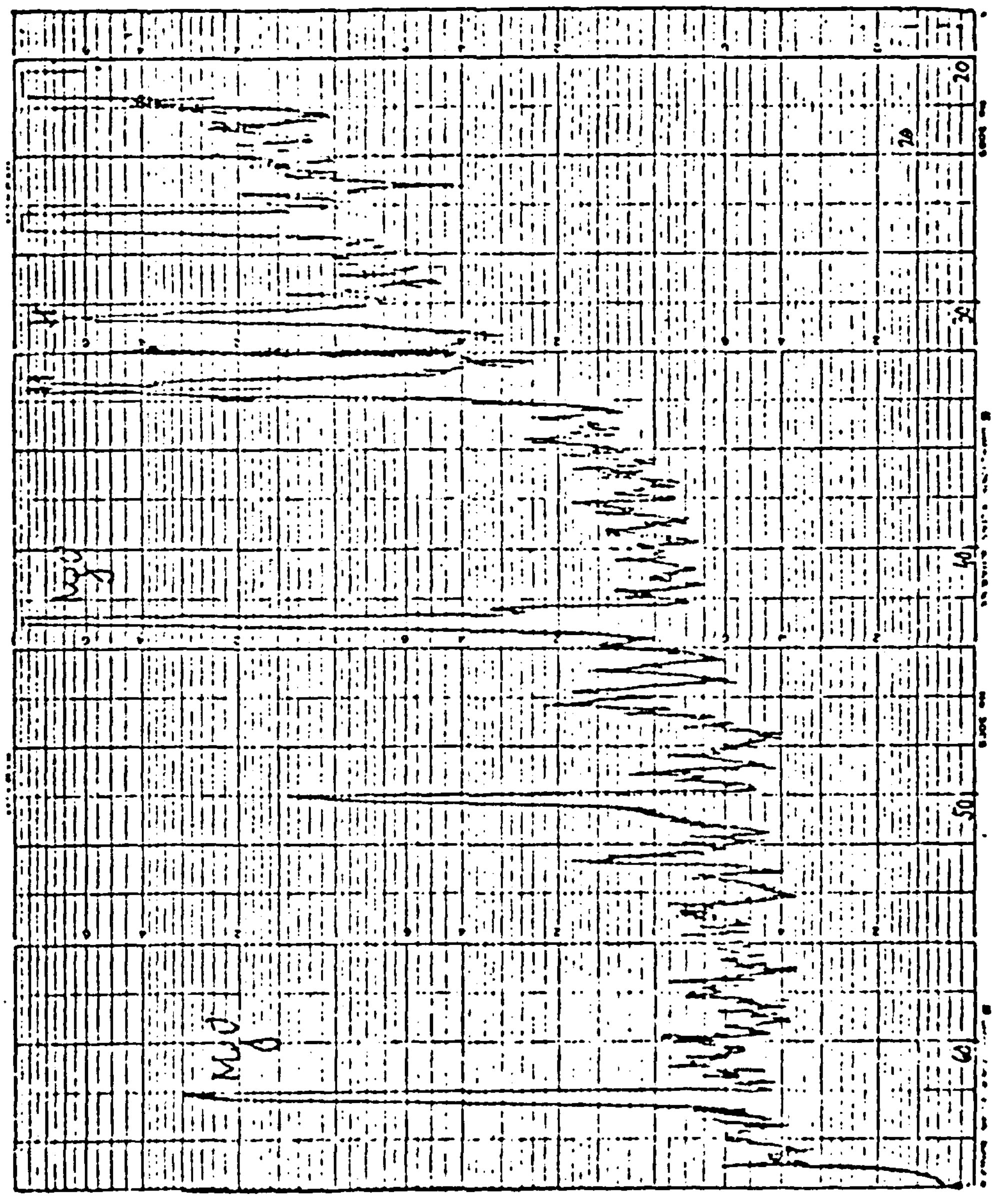

Fig. 95 - X-ray diffraction pattern of SET-45 cold weather mortar wet-cured at 1 month.

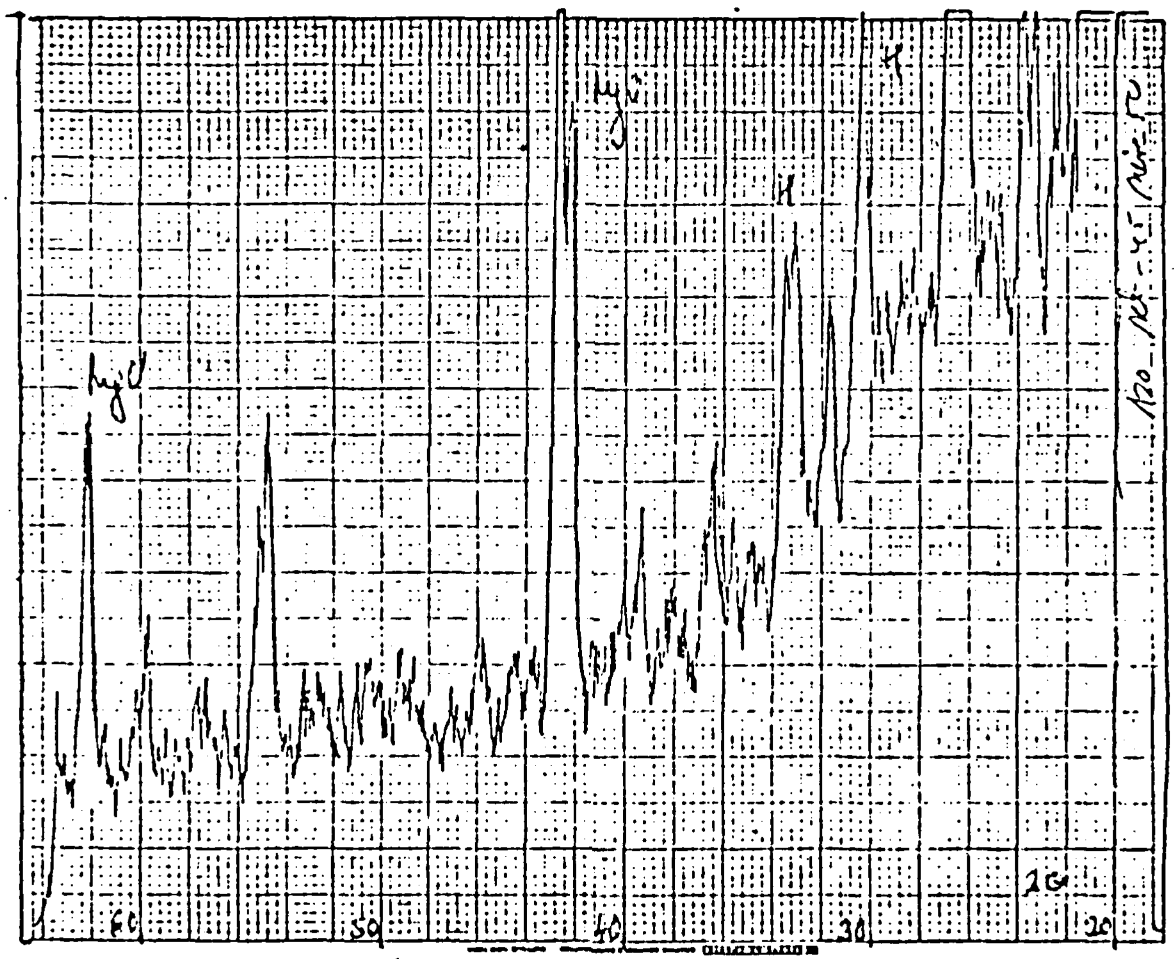

Fig. 96 - X-ray diffraction pattern of SET-45 cold weather mortar dry-cured at 2 months.

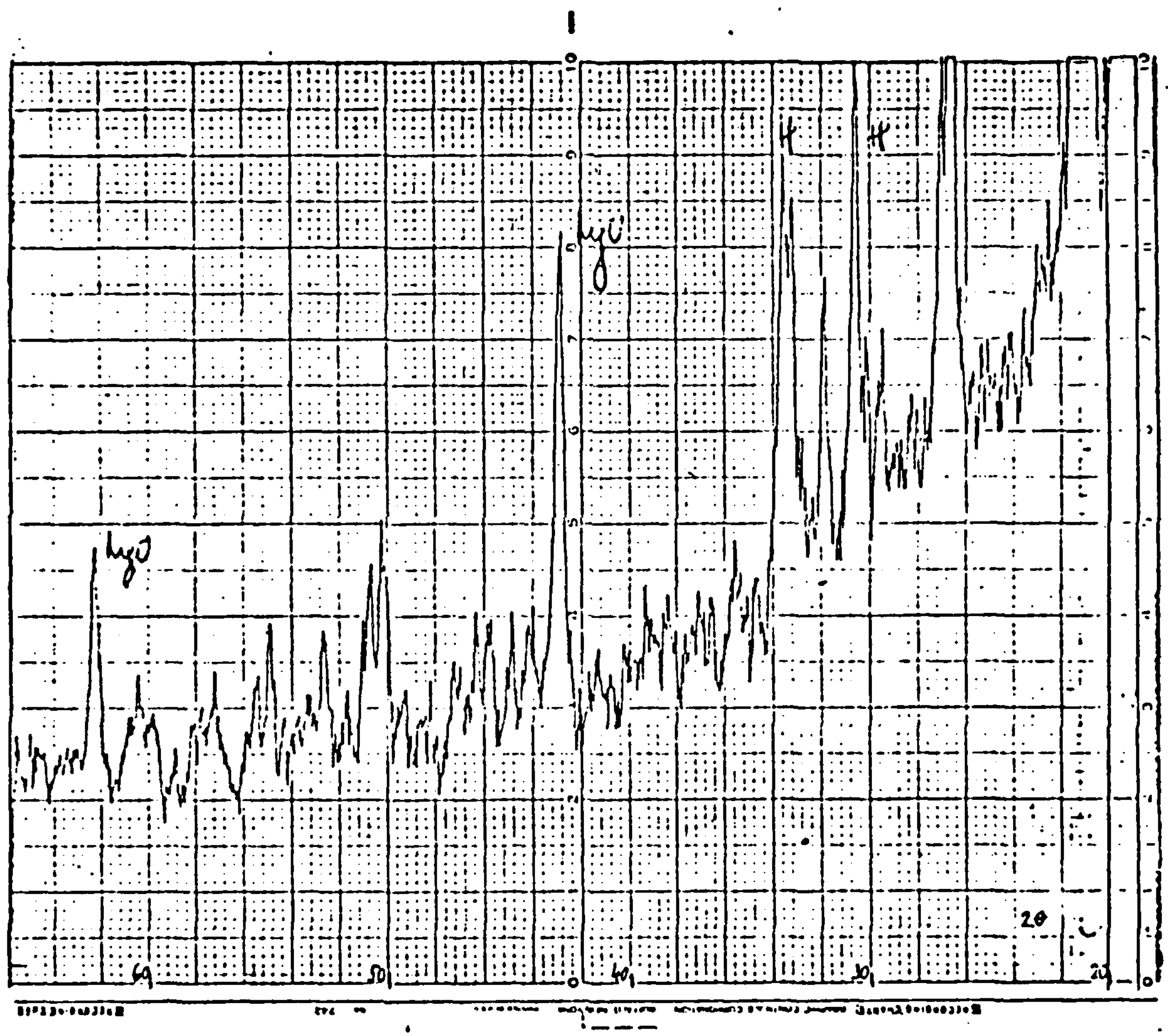

Fig. 97 - X-ray diffraction pattern of SET-45 cold weather mortar wet-cured at 2 months.

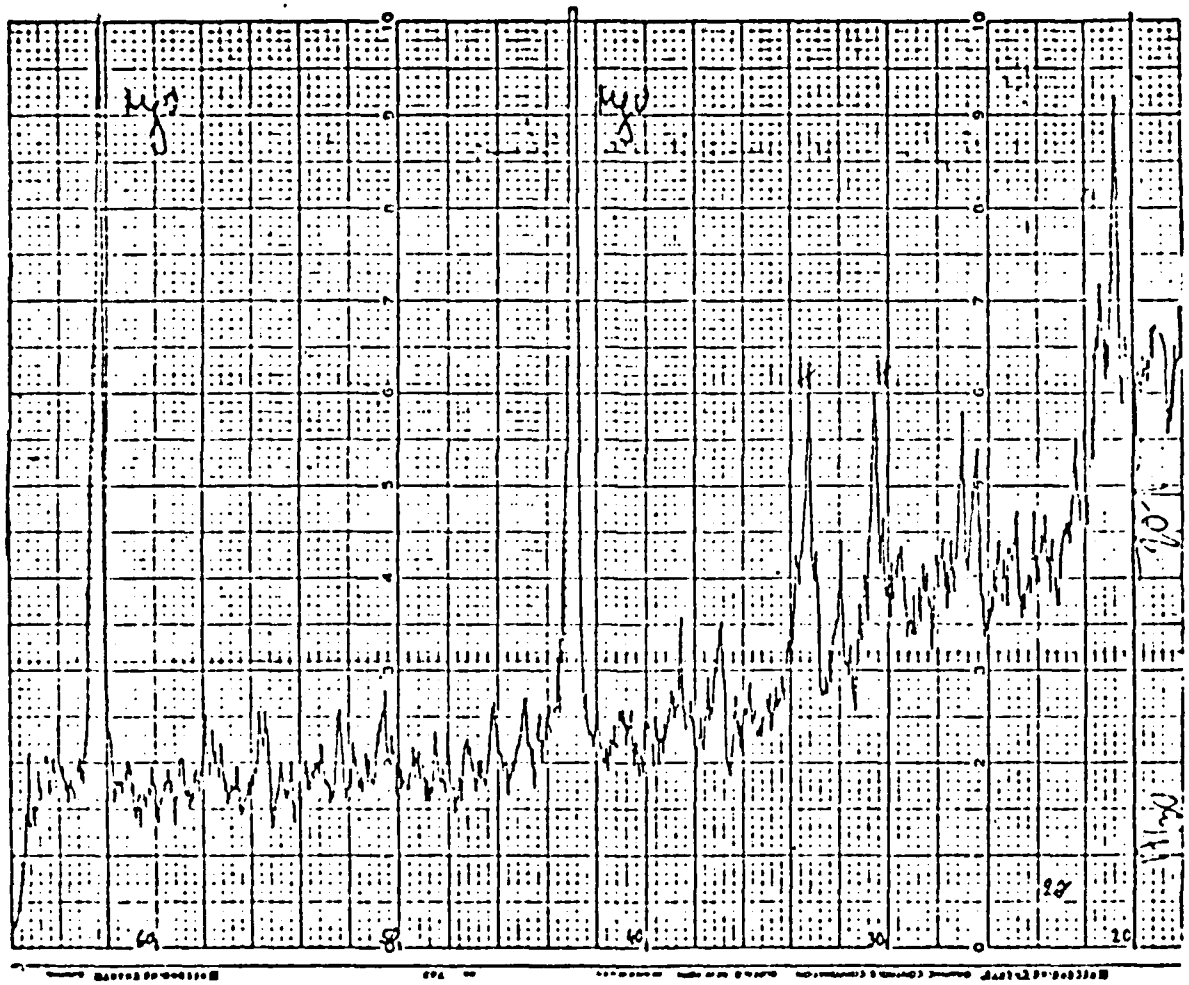

Fig. 98 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 30°F at 3 hours.

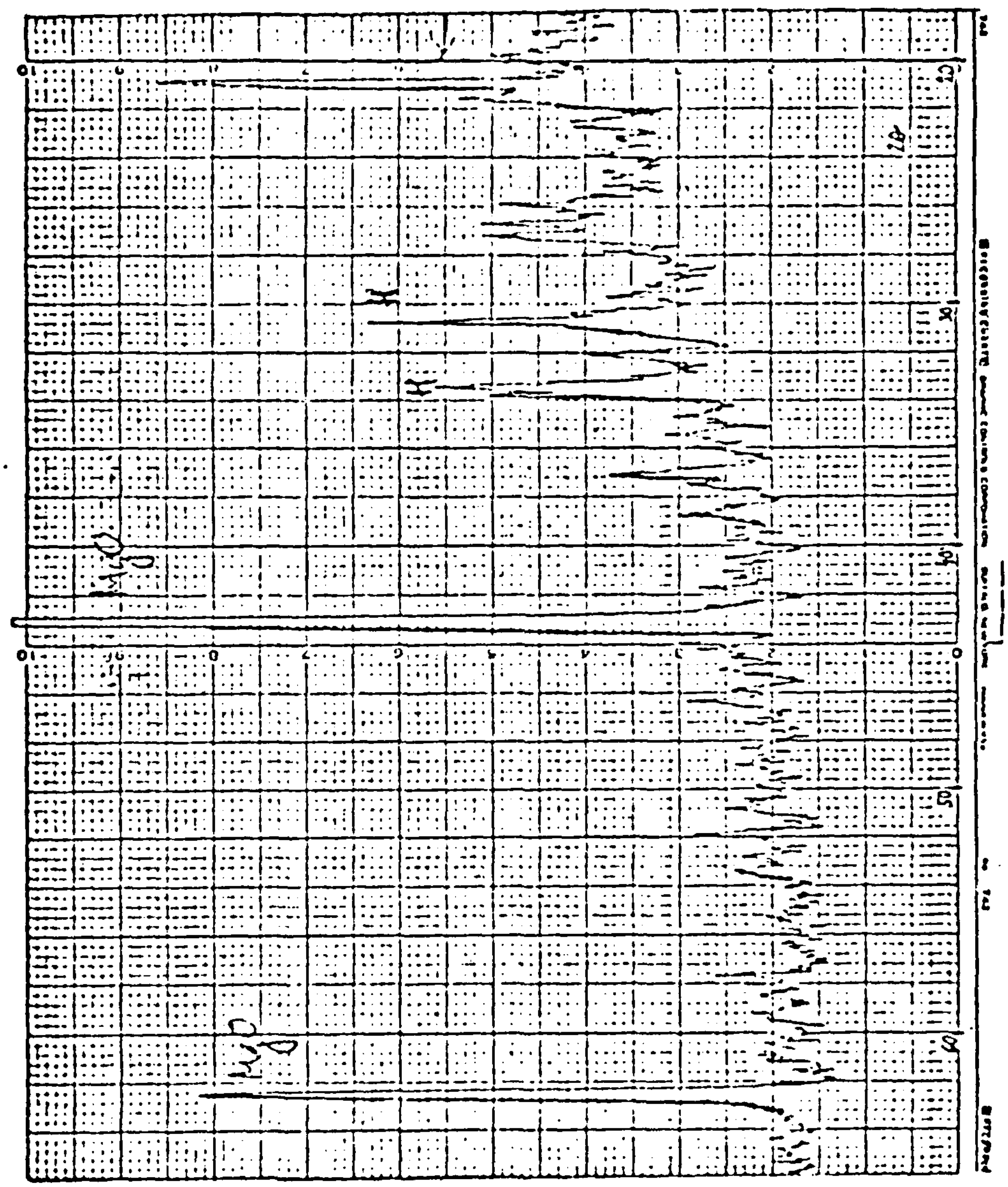

Fig. 99 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 30°F at 1 day.

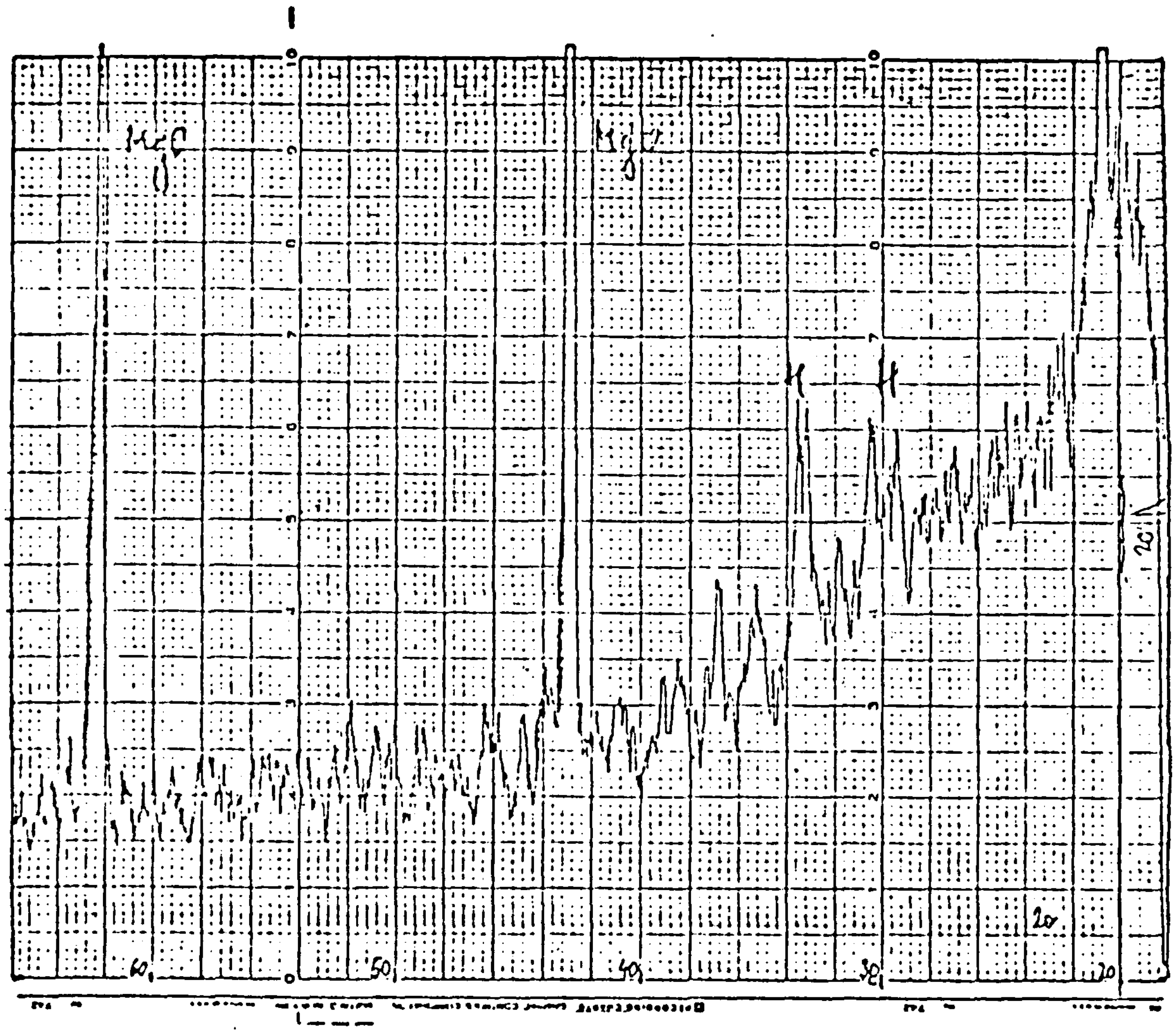

Fig. 100 - X-ray diffraction pattern of SET-45 cold weather paste mixed and cured at 30°F at 3 days.

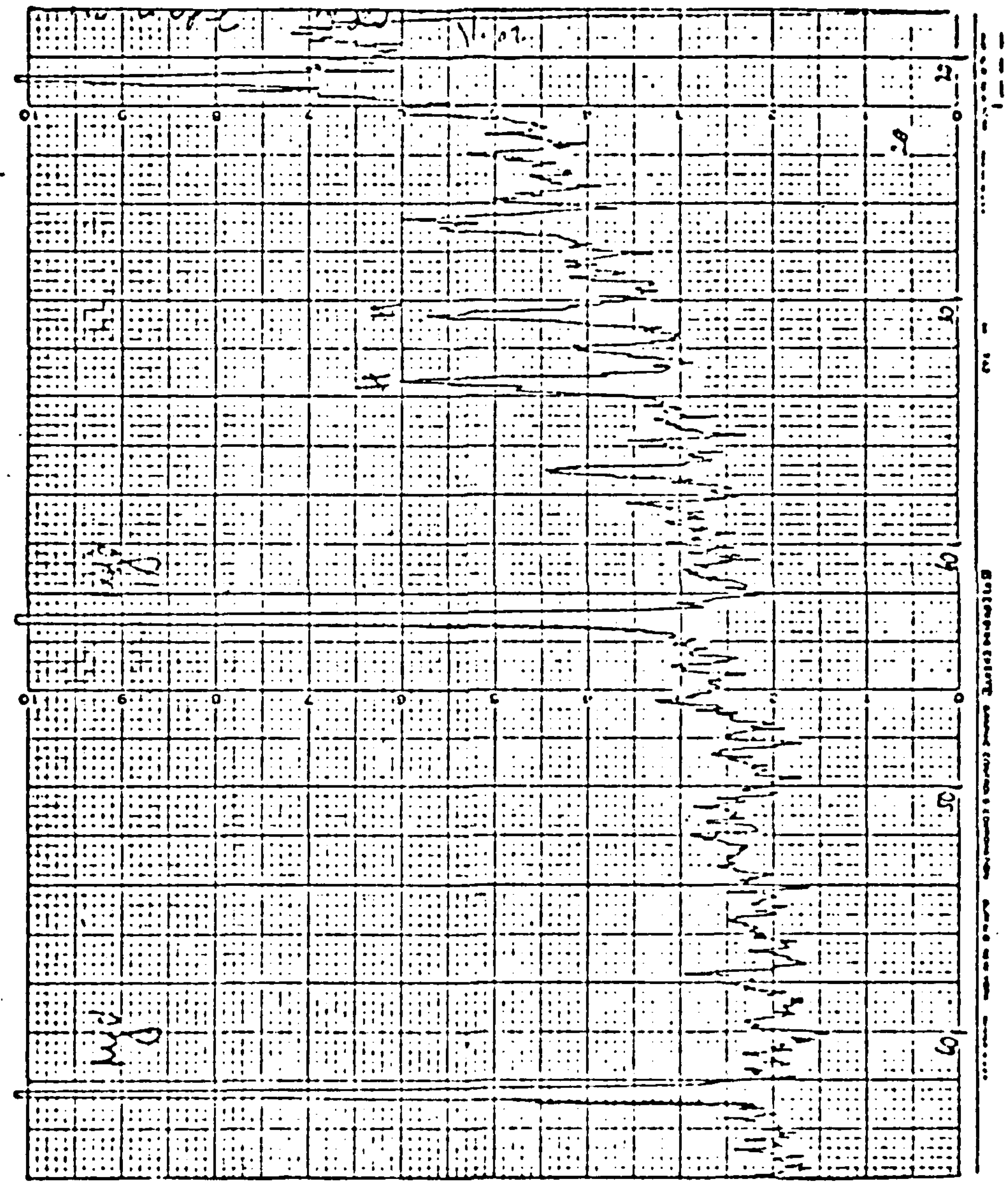

Fig. 101 - X-ray diffraction of SET-45 cold weather paste mixed and cured at 30°F at 7 days.

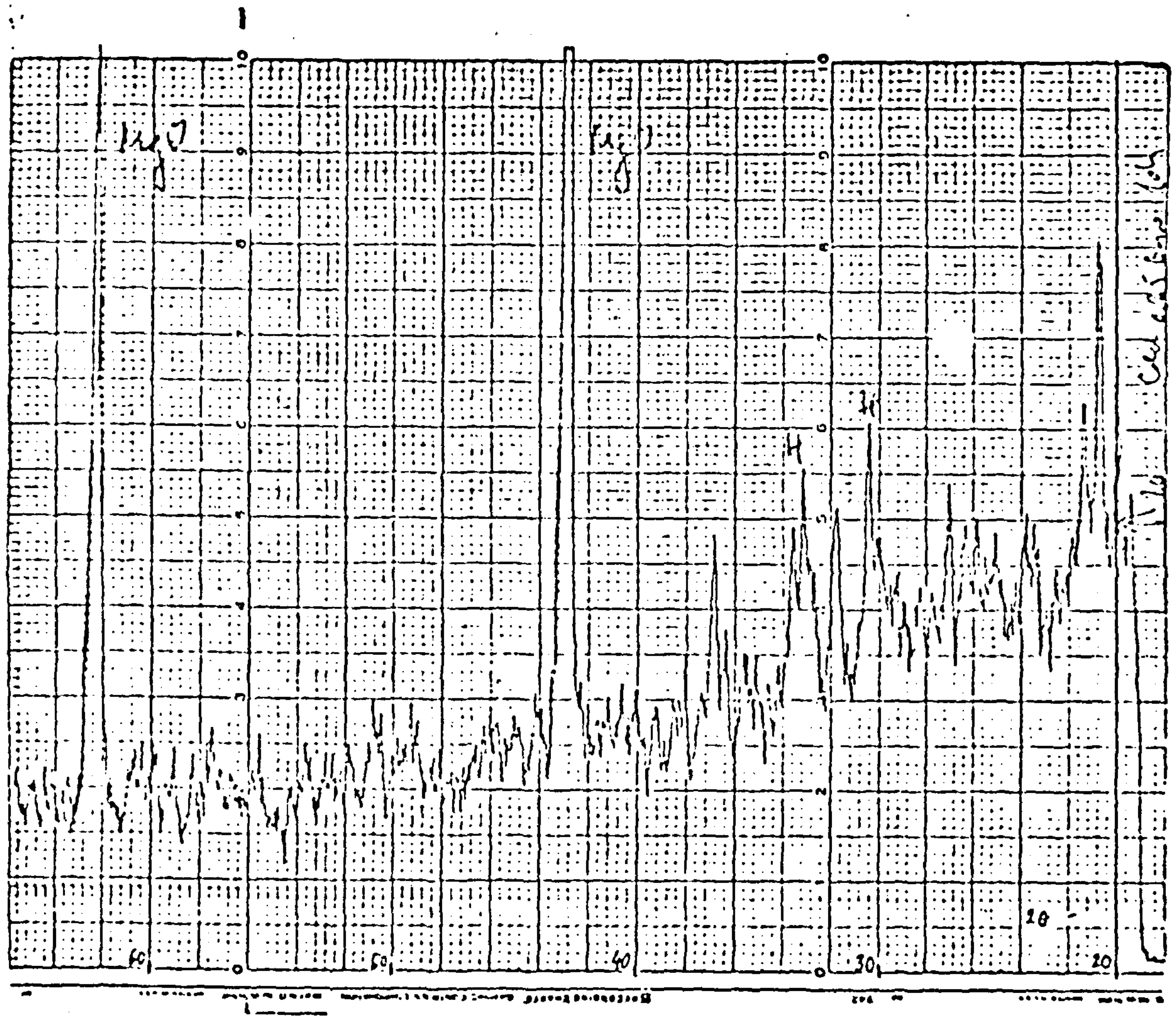

Fig. 102 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed and cured at 30°F at 1 day.

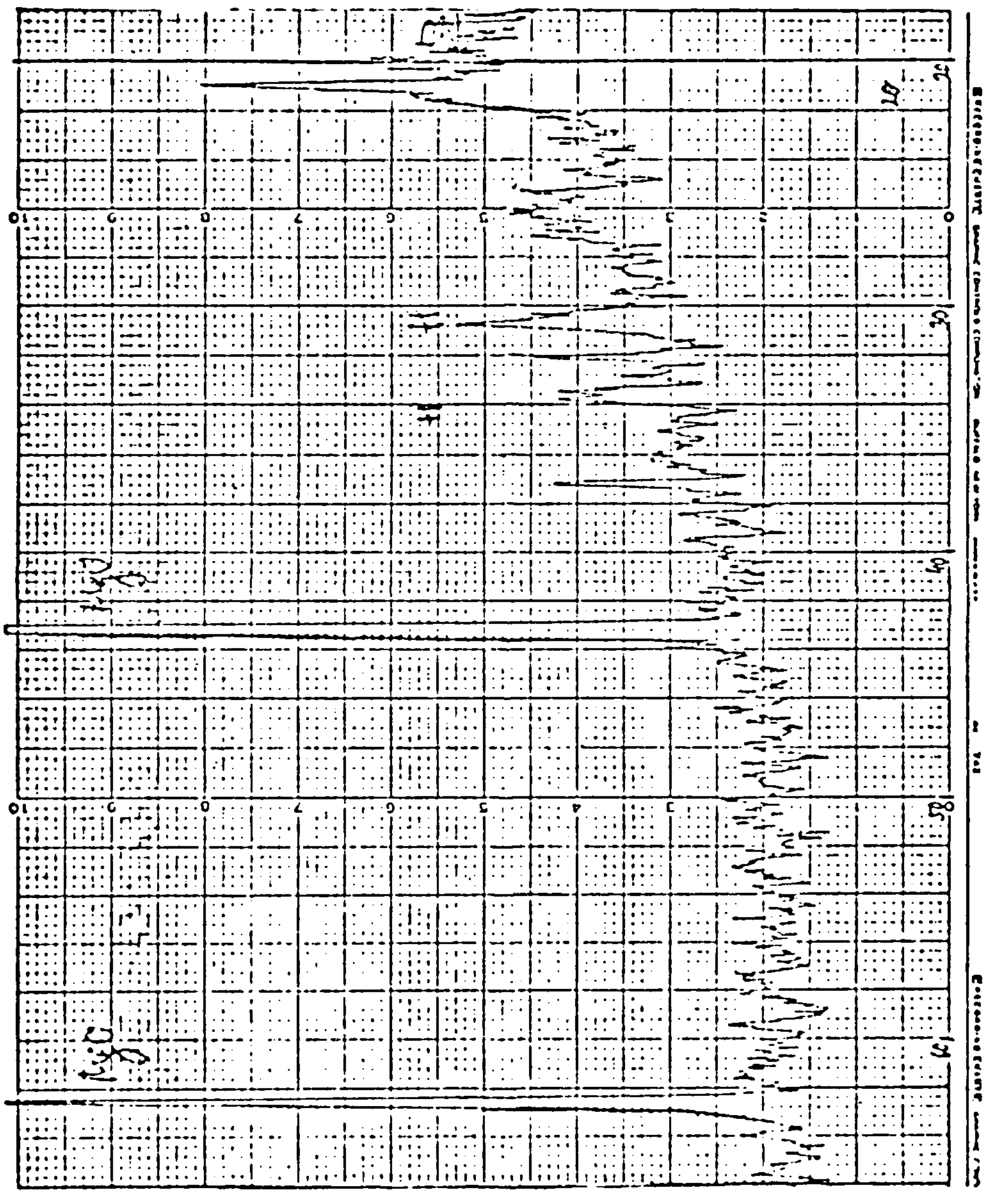

Fig. 103 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed and cured at 30°F at 3 days.

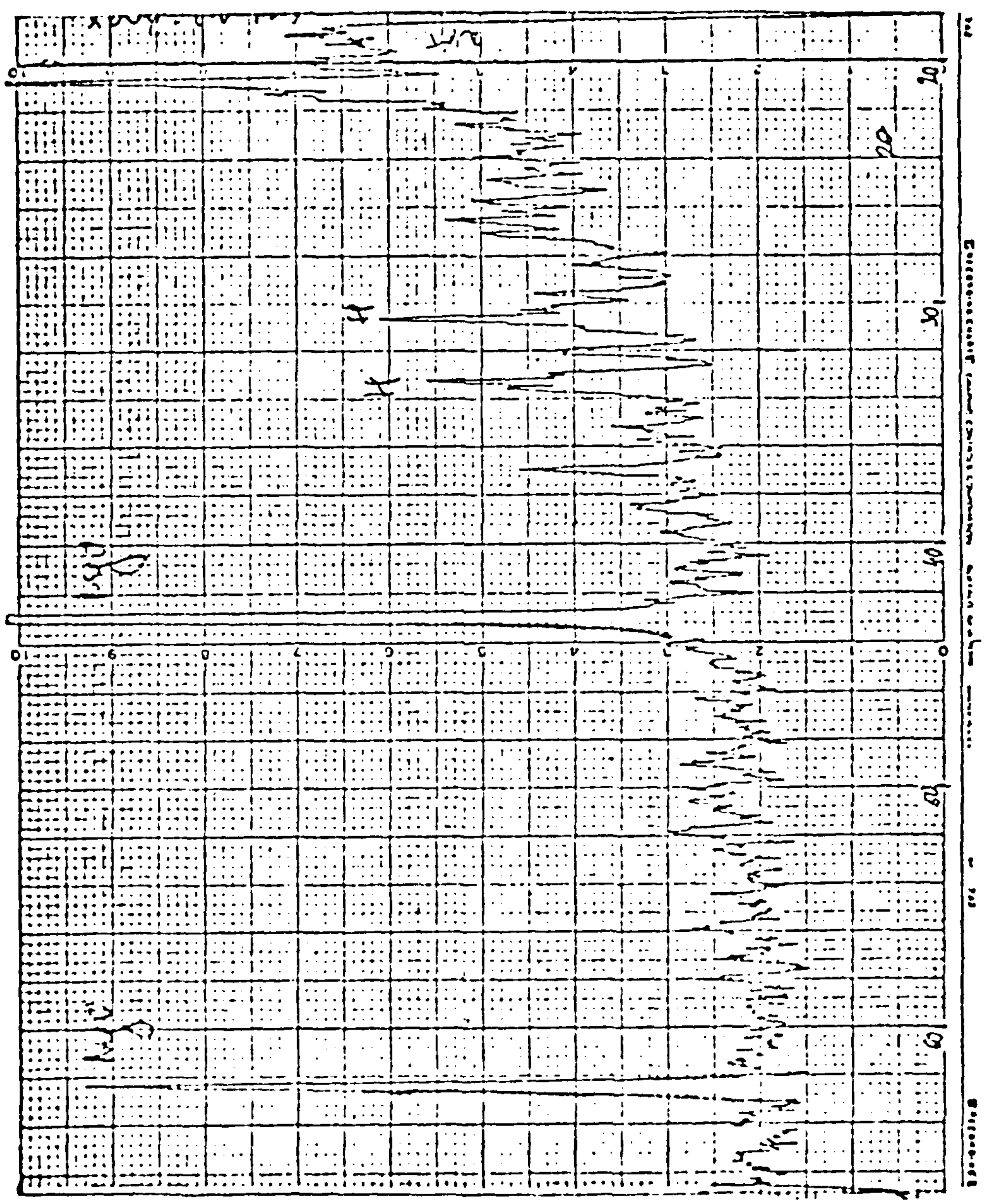

Fig. 104 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed and cured at 30°F at 7 days.

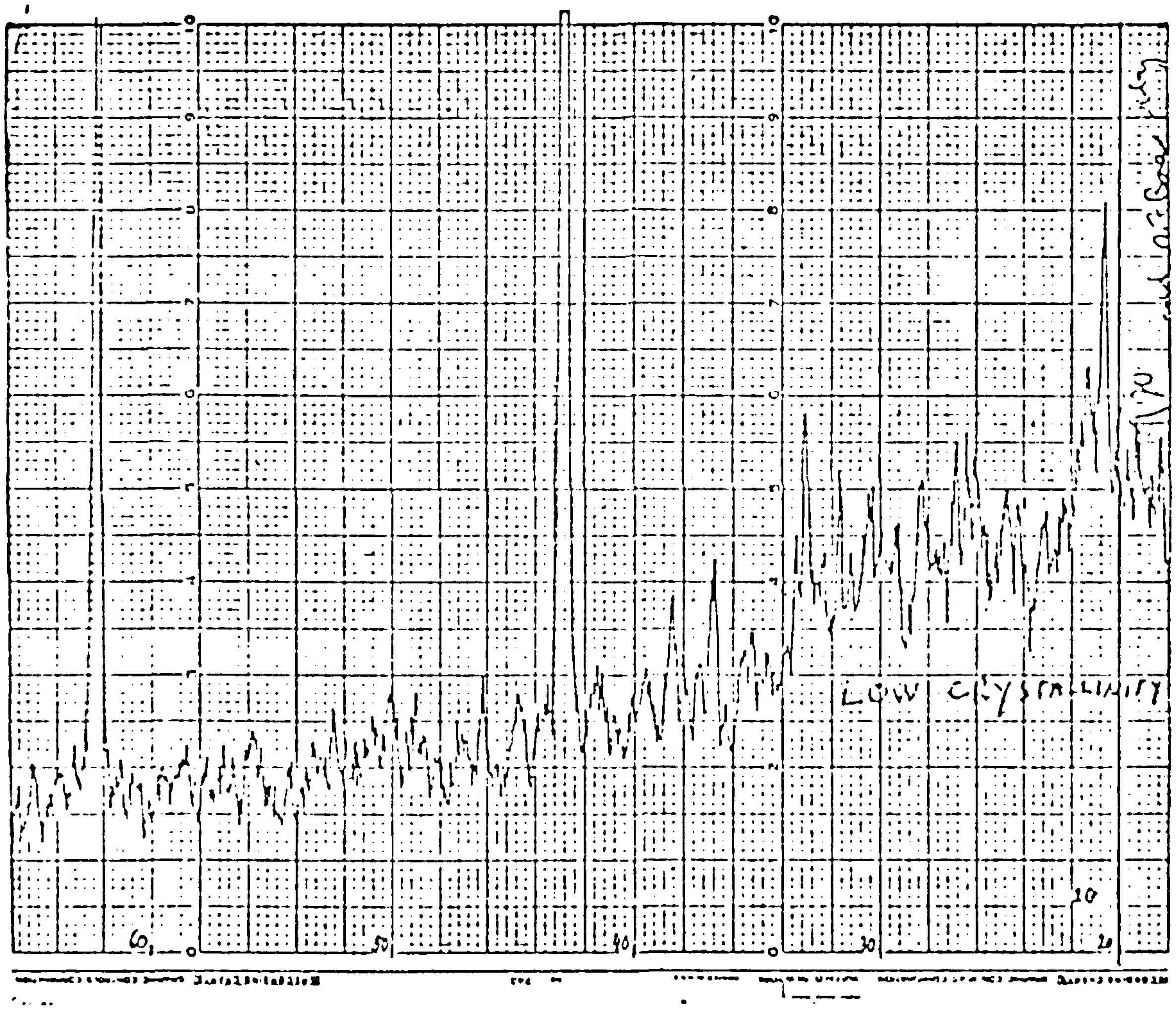

Fig. 105 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 30°F at 1 day.

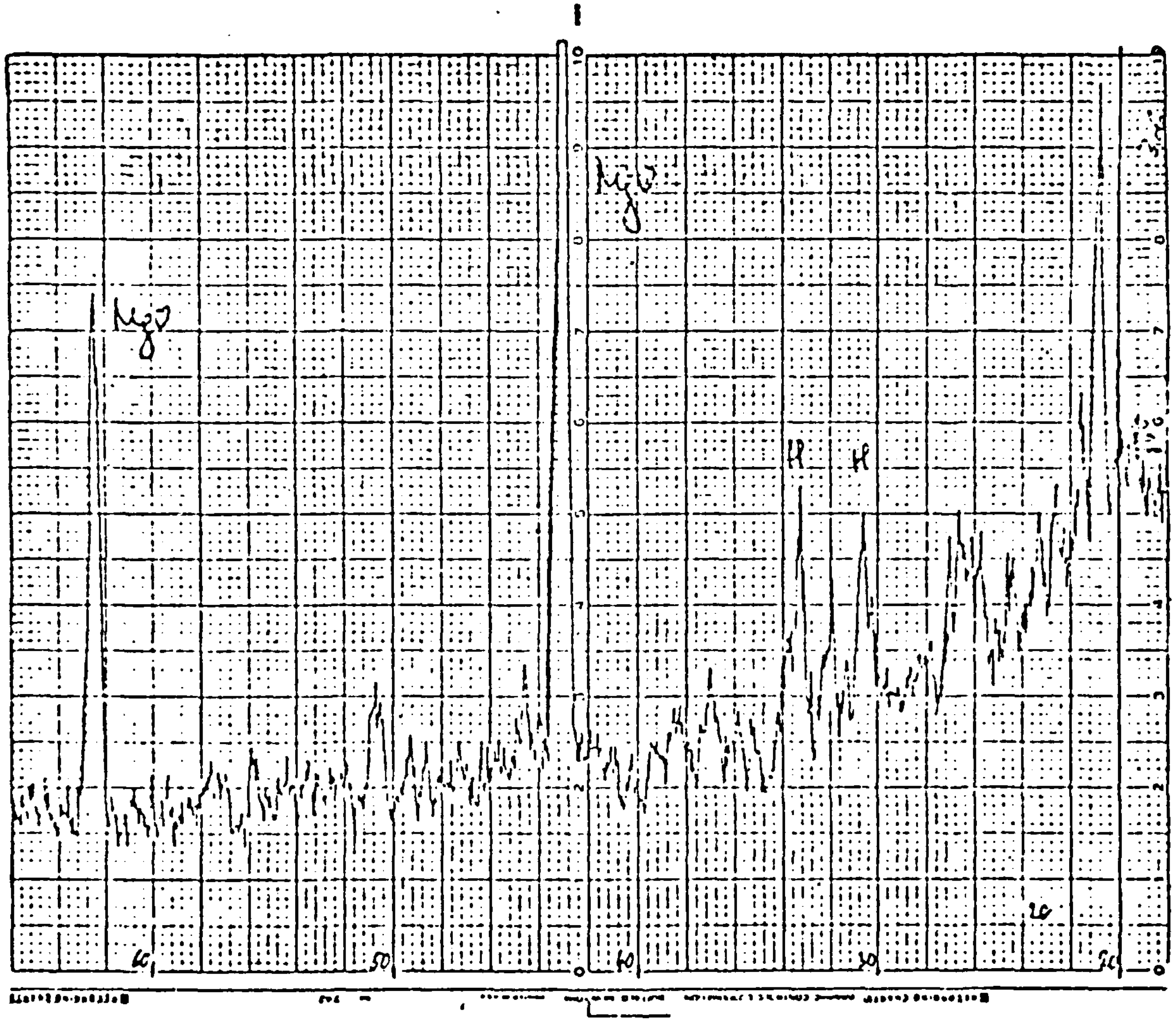

Fig. 106 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 30°F at 3 days.

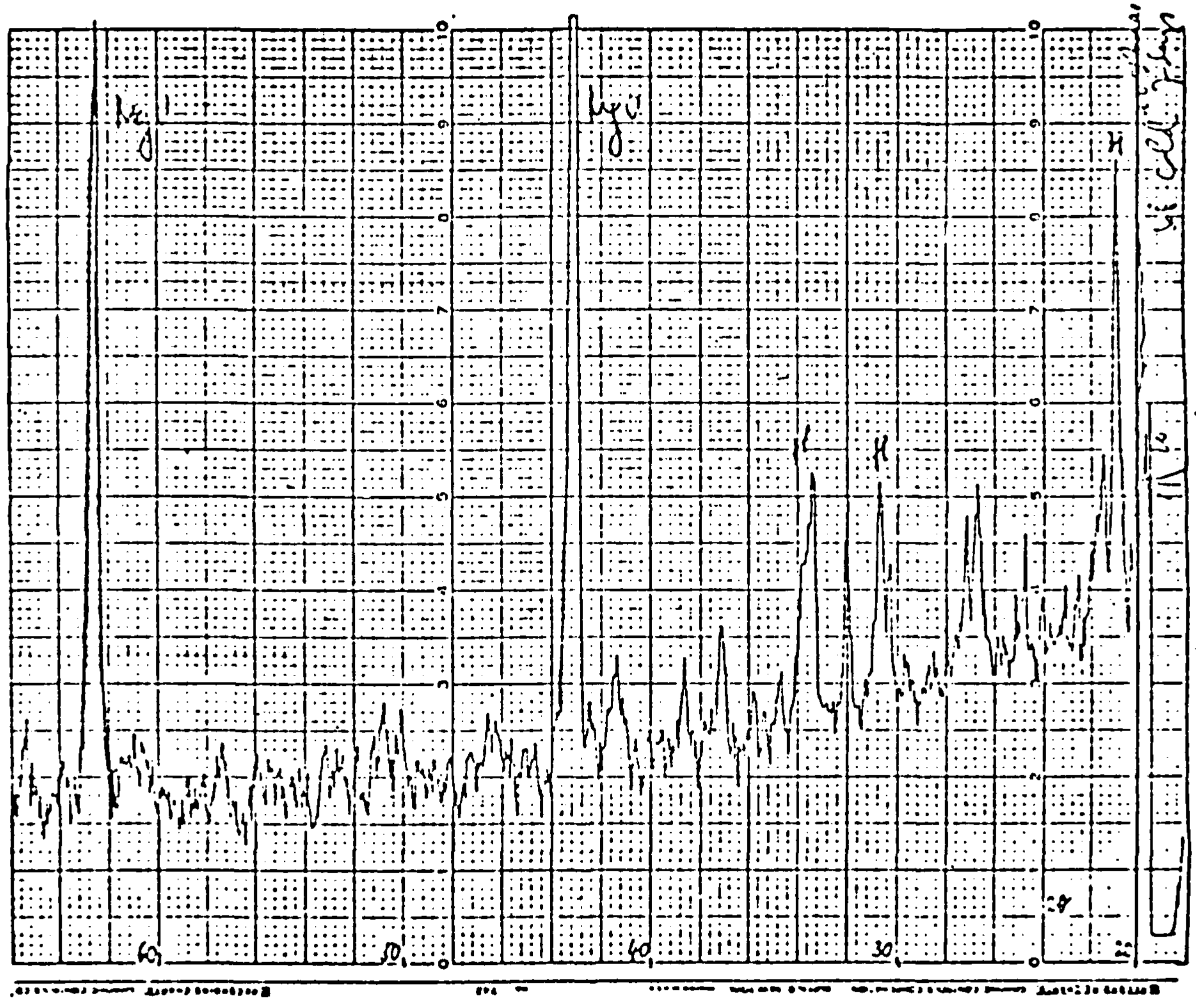

Fig. 107 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed and cured at 30°F at 7 days.

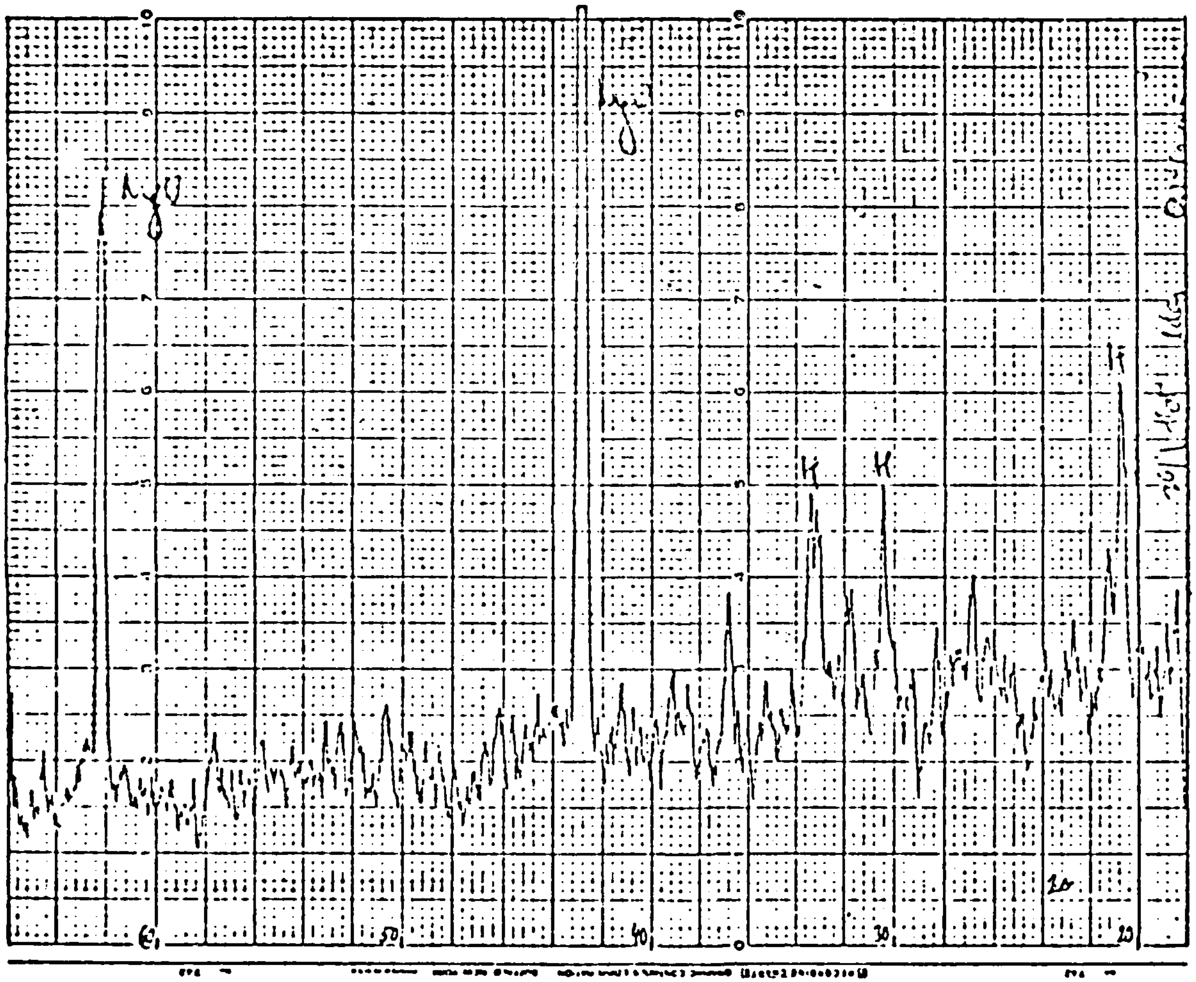

Fig. 108 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 30°F at 1 day.

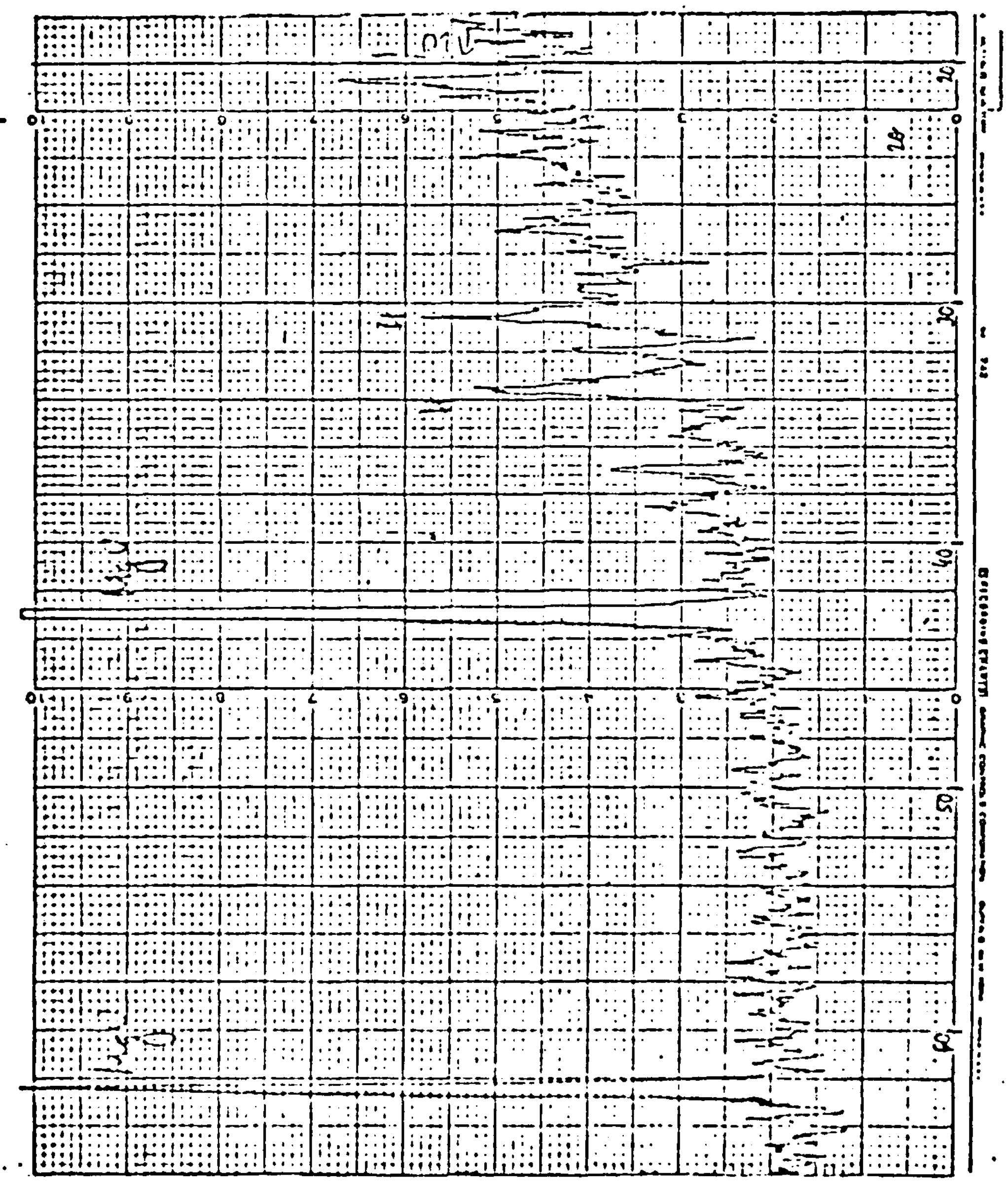

Fig. 109 - X-ray diffraction pattern of SET-45 hot weather paste mixed and cured at 30°F at 3 days.

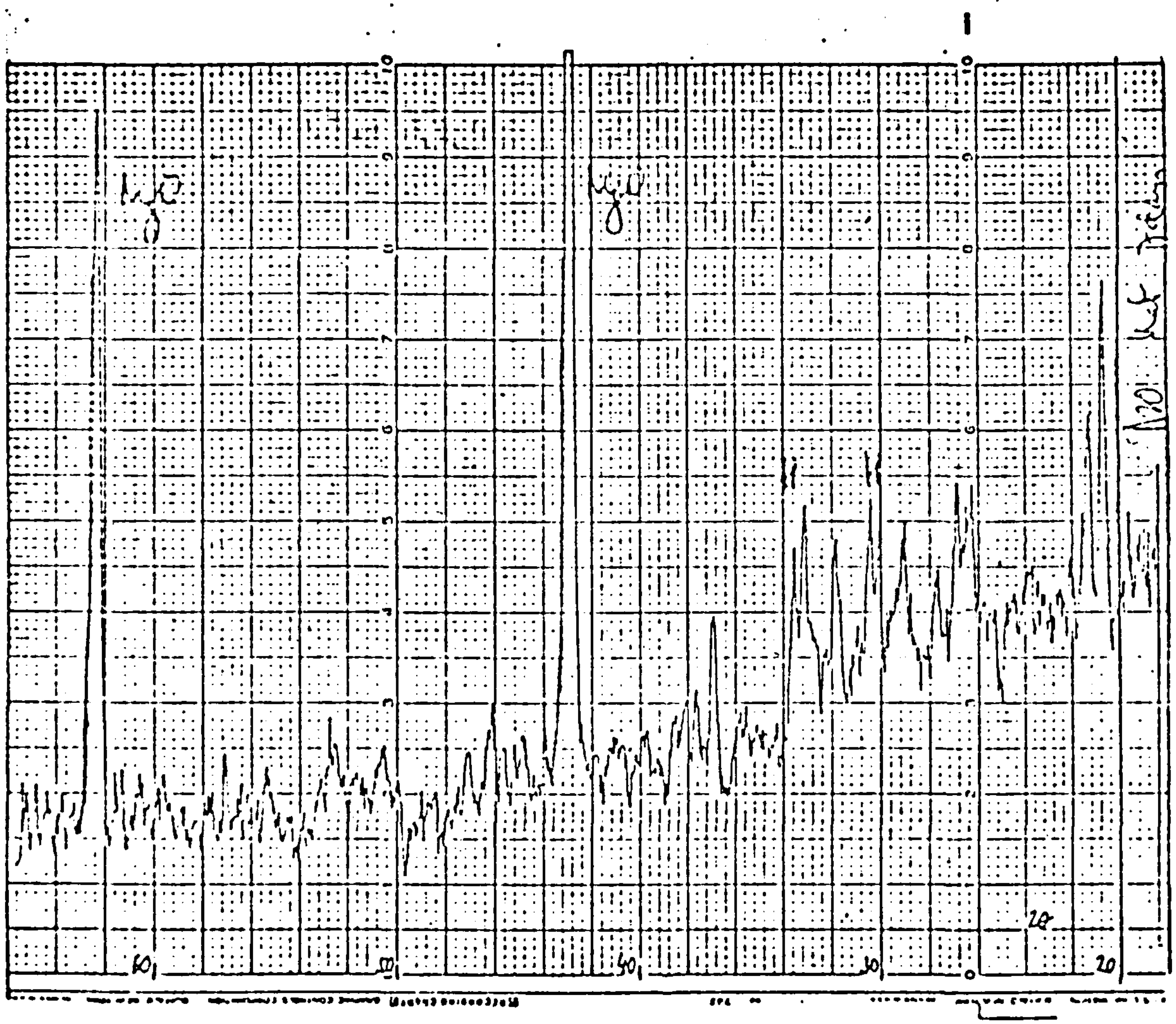

Fig. 110 - X-ray diffraction pattern of SET-45 hot weather paste mixed and
cured at 30°F at 7 days.

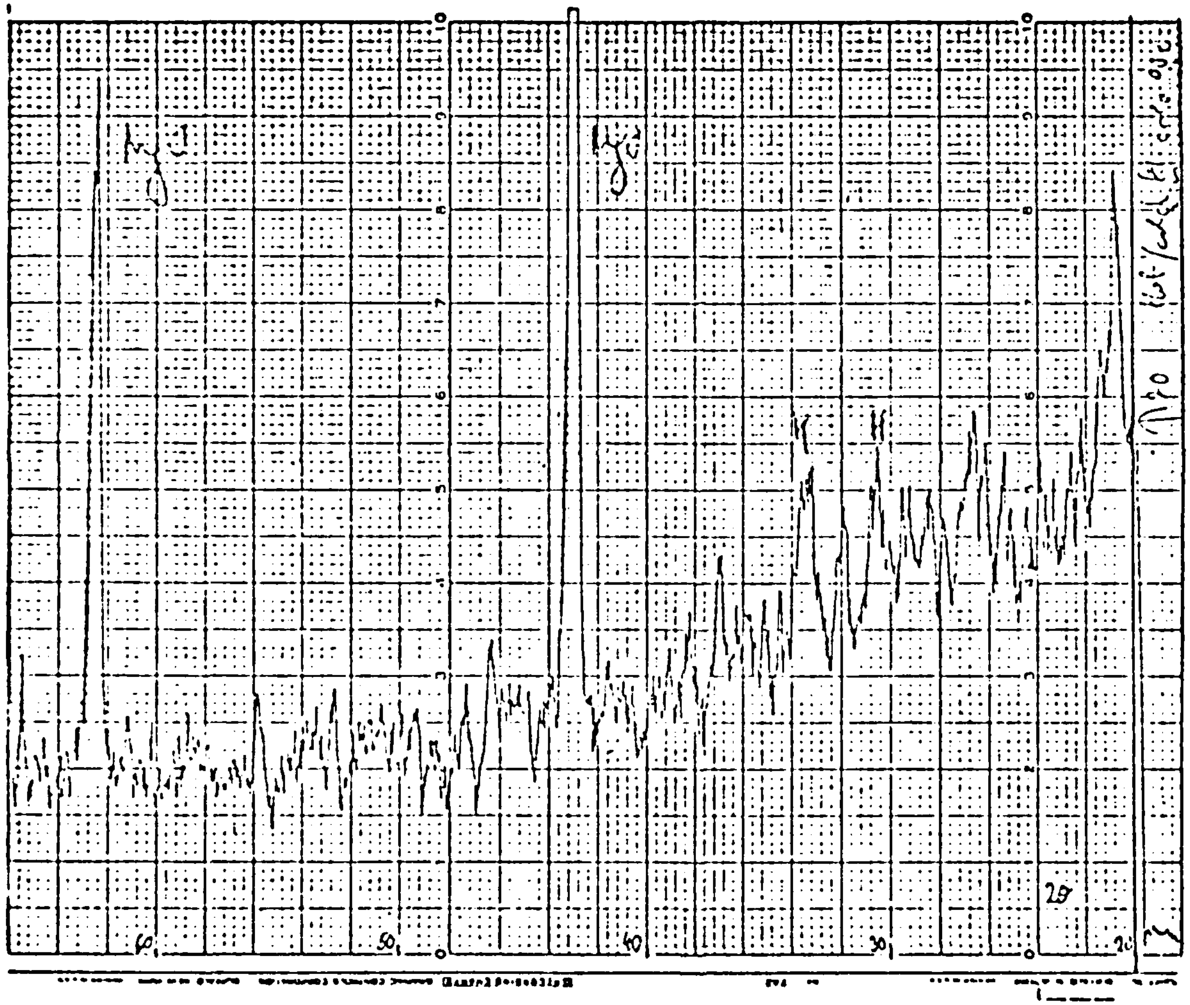

Fig. 111 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 30°F at 3 hours.

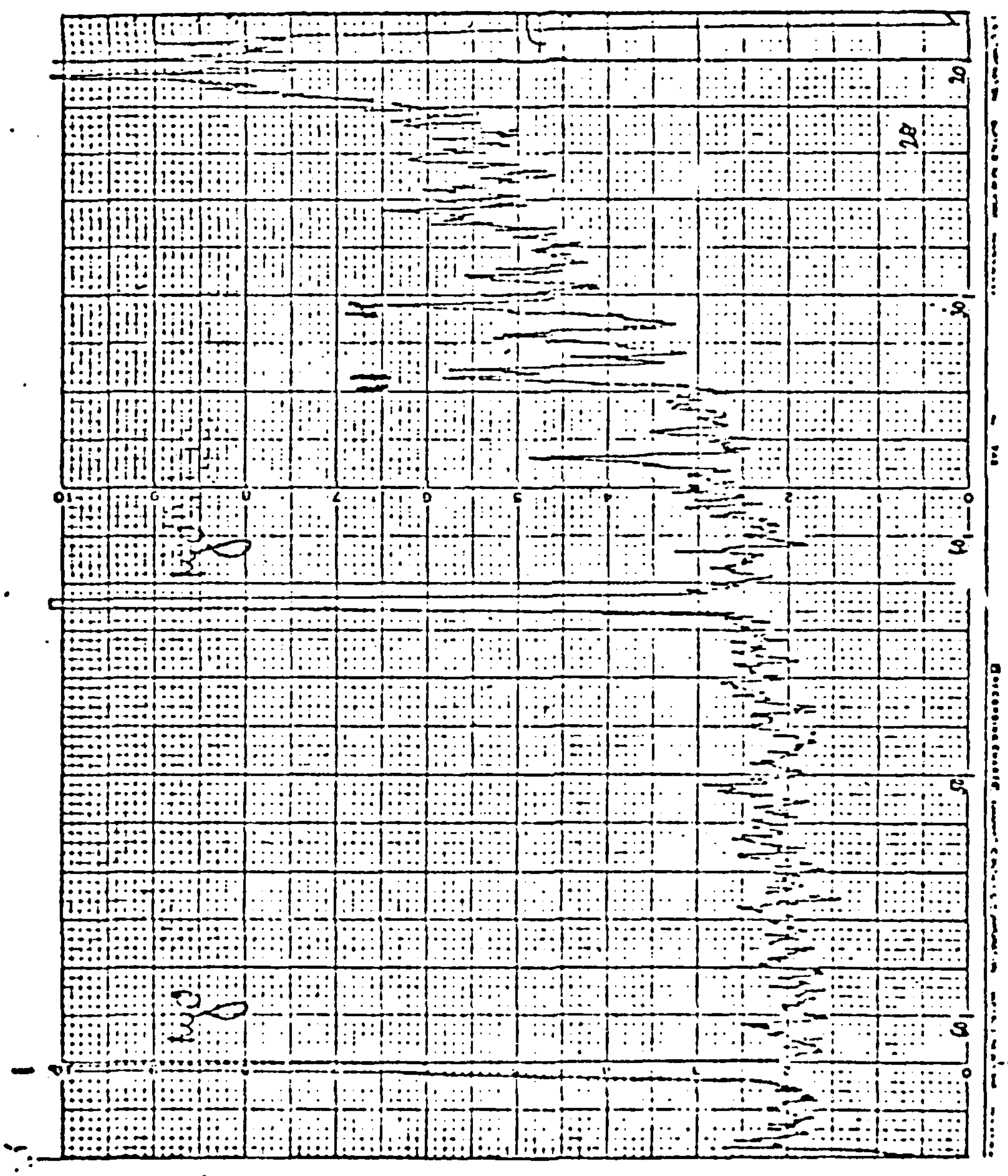

Fig. 112 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 30°F at 1 day.

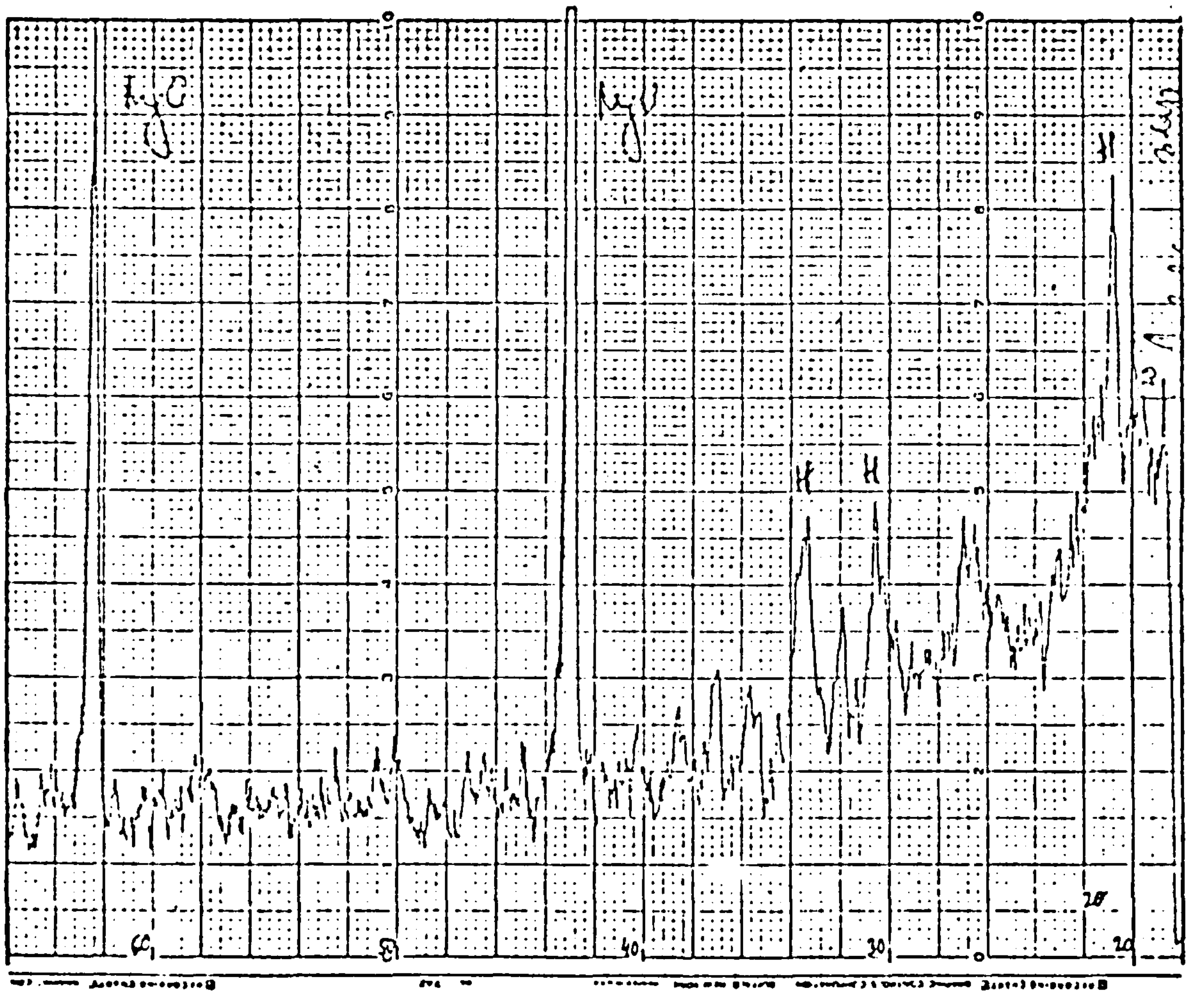

Fig. 113 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 30°F at 3 days.

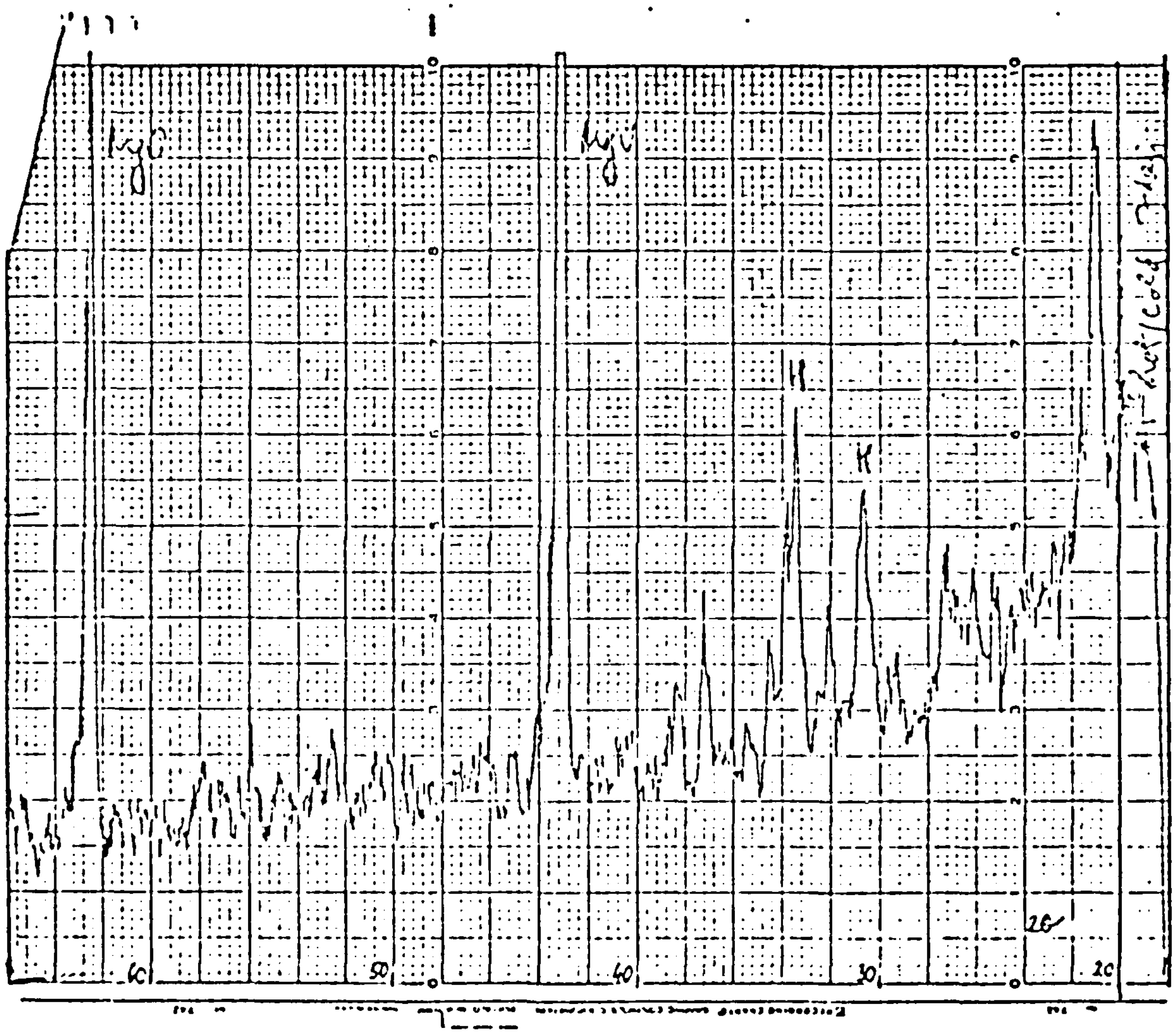

Fig. 114 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed and cured at 30°F at 7 days.

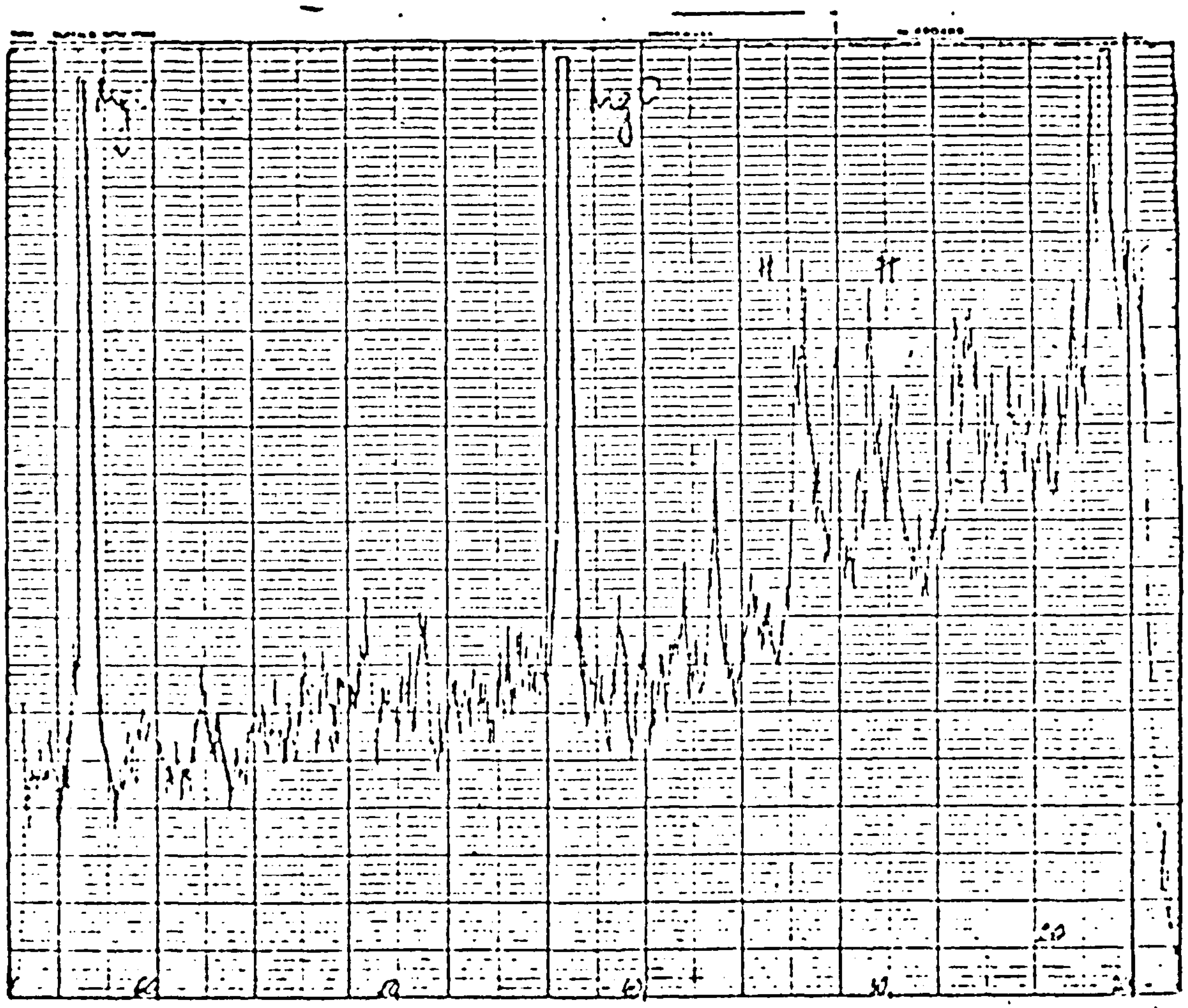

Fig. 115 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 100°F and cured at 30°F at 1 hour.

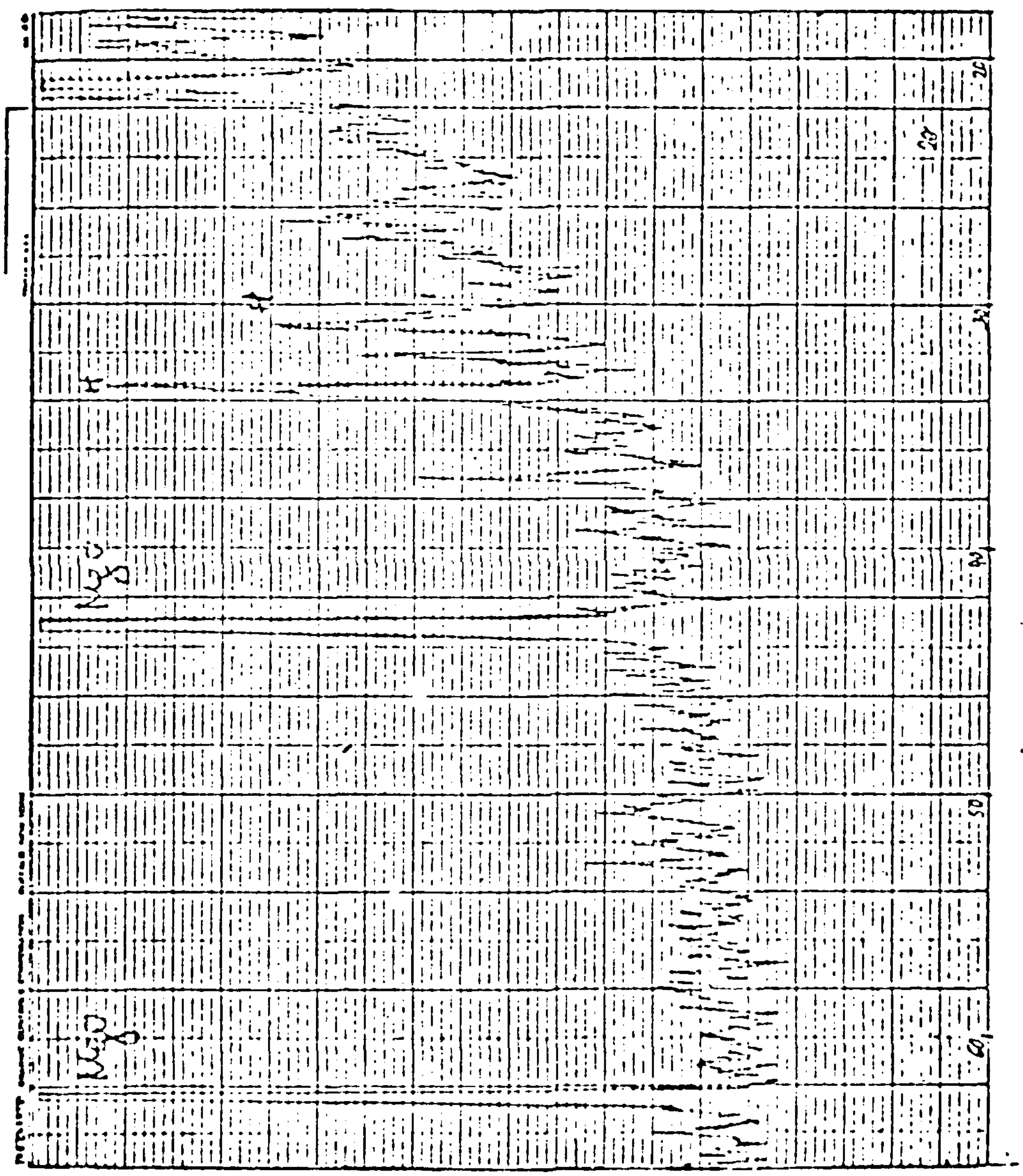

Fig. 116 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 100°F and cured at 30°F at 3 hours.

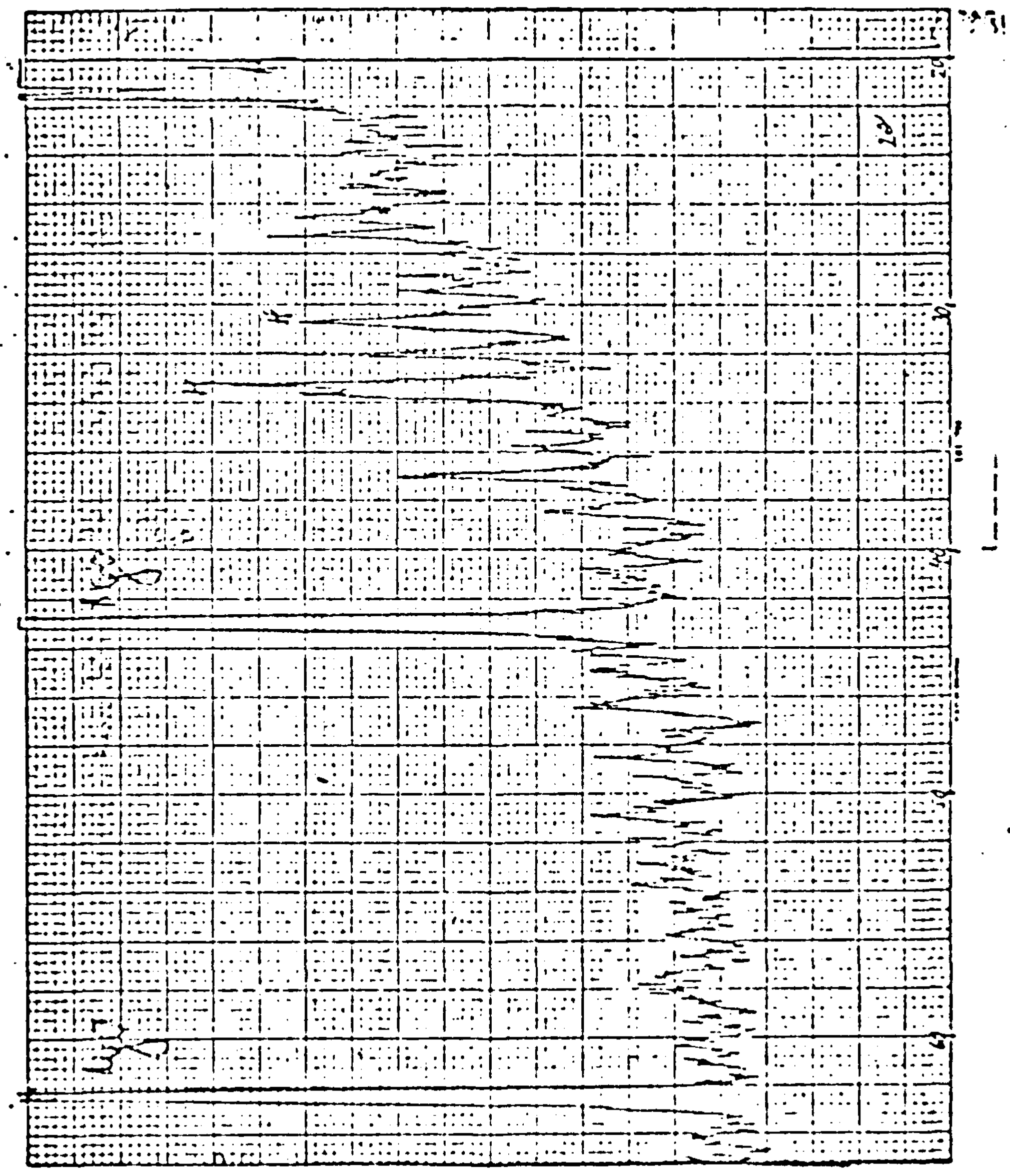

Fig. 117 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 100°F and cured at 30°F at 1 day.

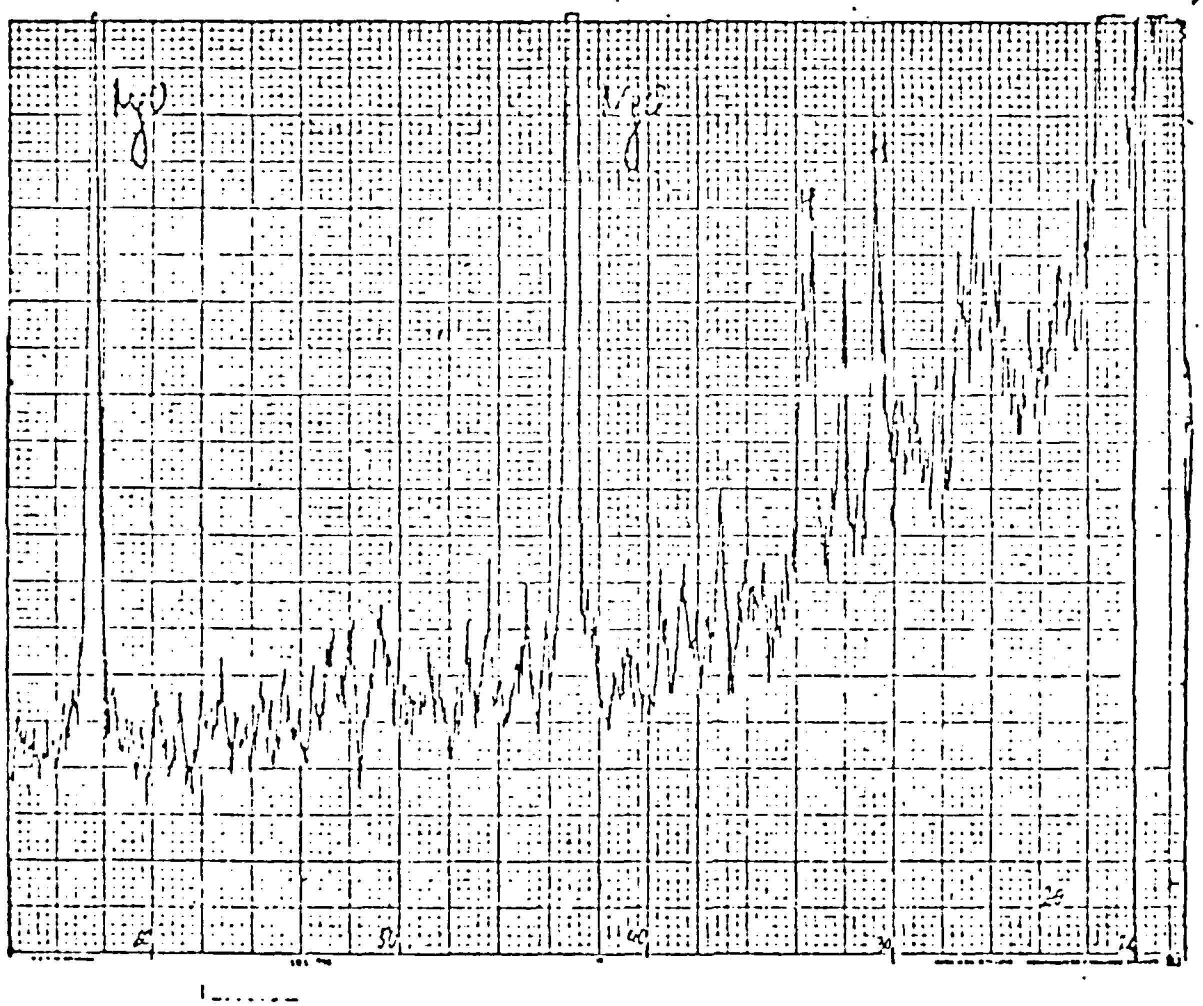

Fig. 118 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 100°F and cured at 30°F at 7 days.

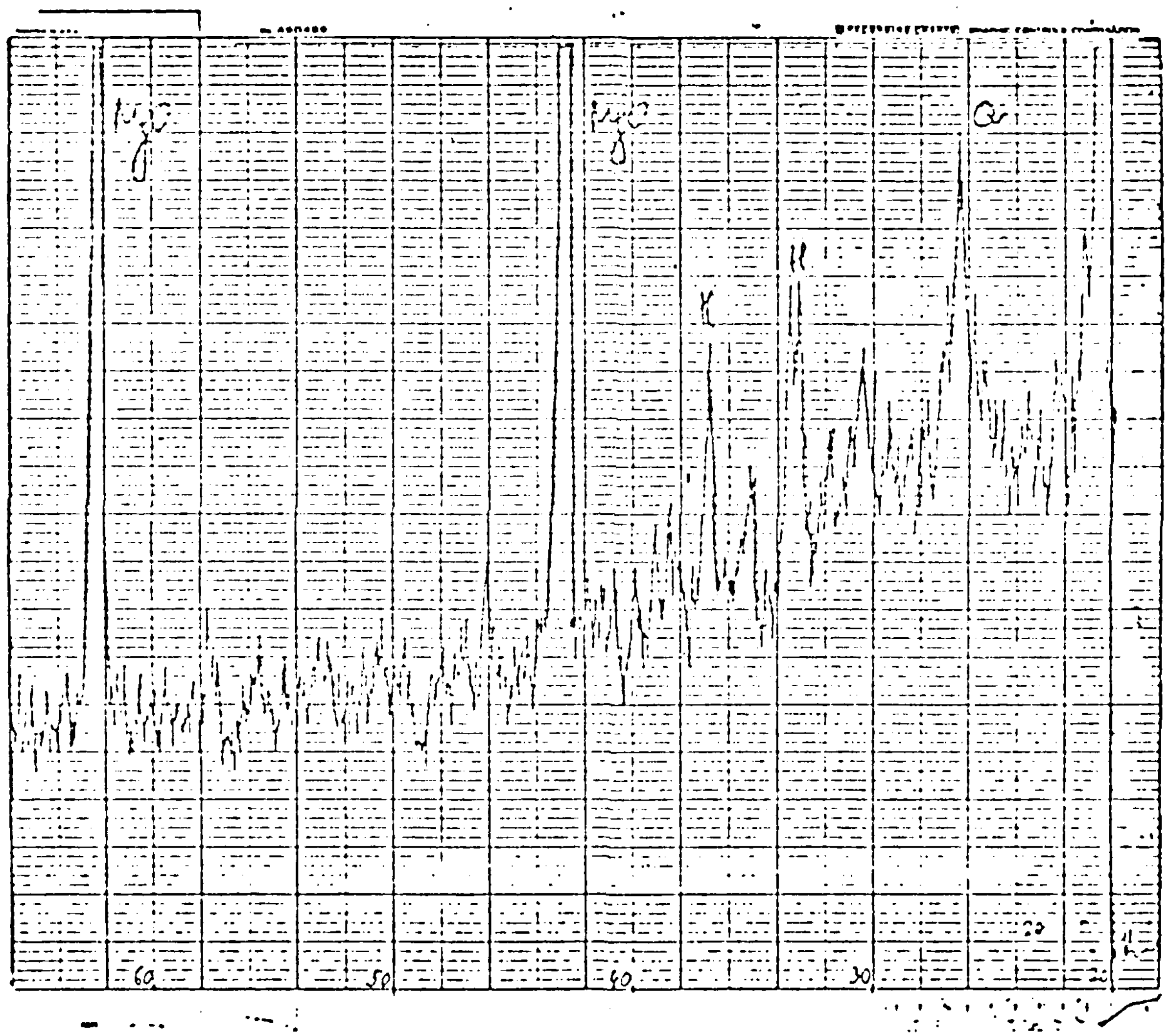

Fig. 119 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed at 100°F and cured at 30°F at 3 hours.

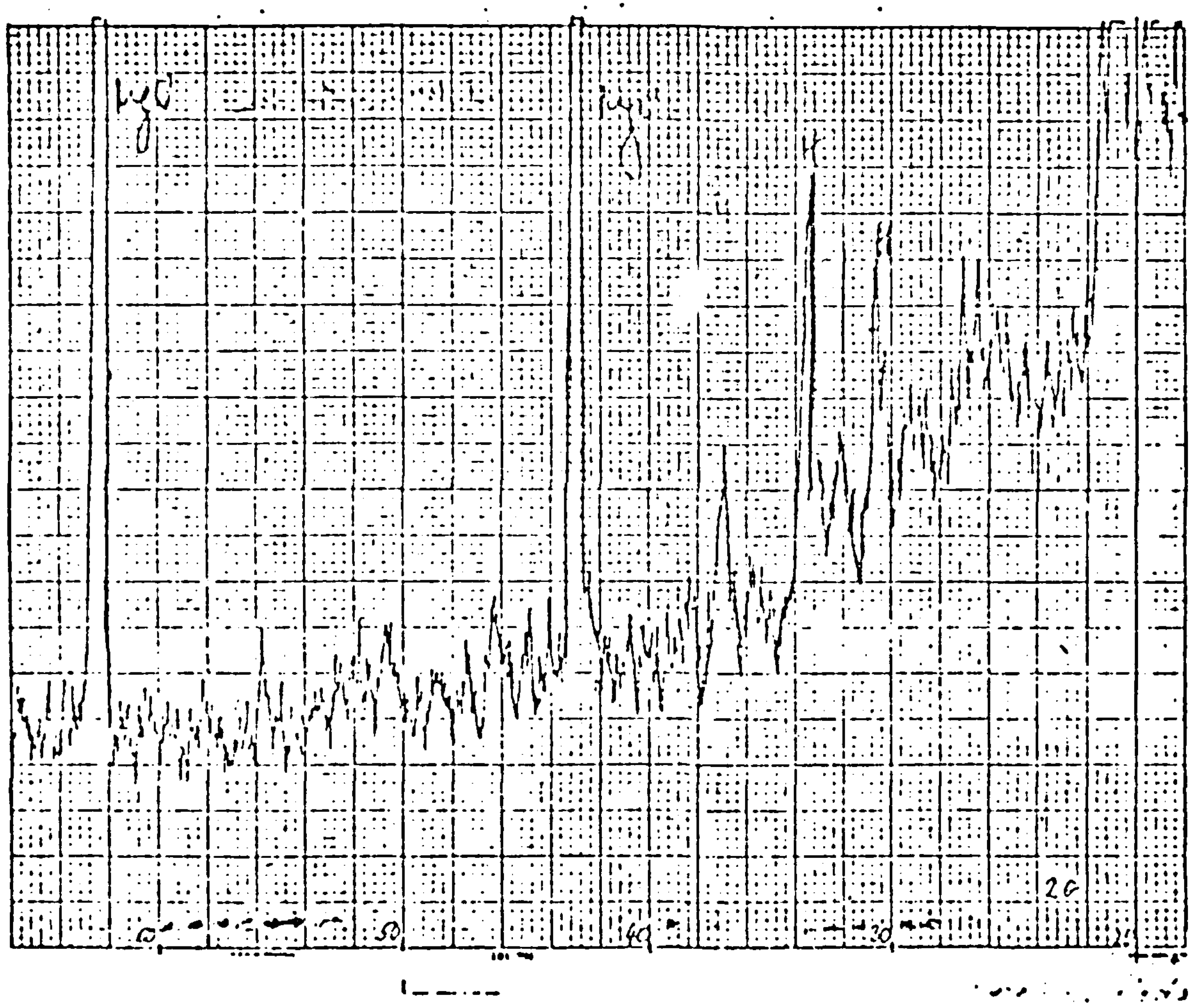

Fig. 120 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed at 100°F and cured at 30°F at 1 day.

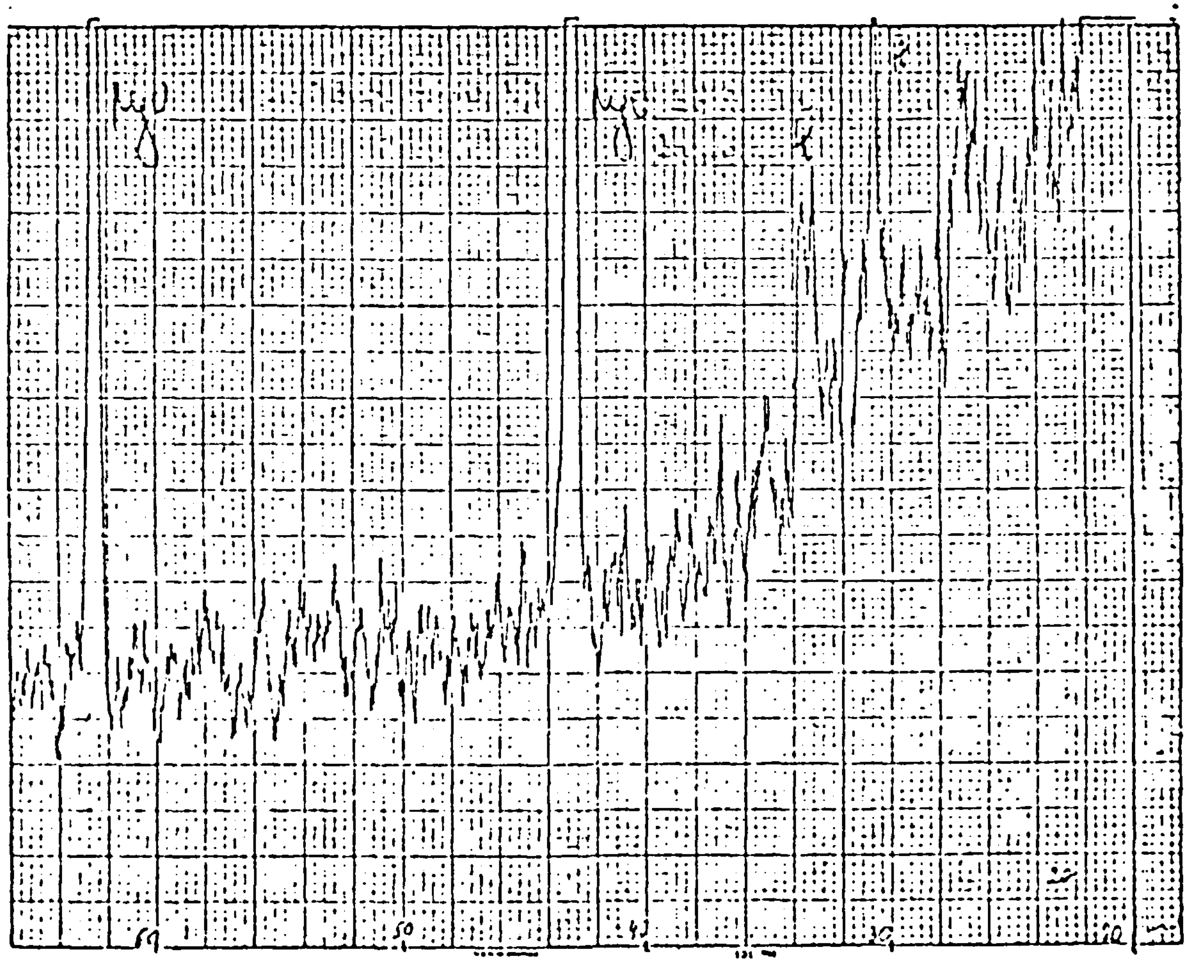

Fig. 121 - X-ray diffraction pattern of SET-45 cold weather paste with 1.7% borax mixed at 100°F and cured at 30°F at 7 days.

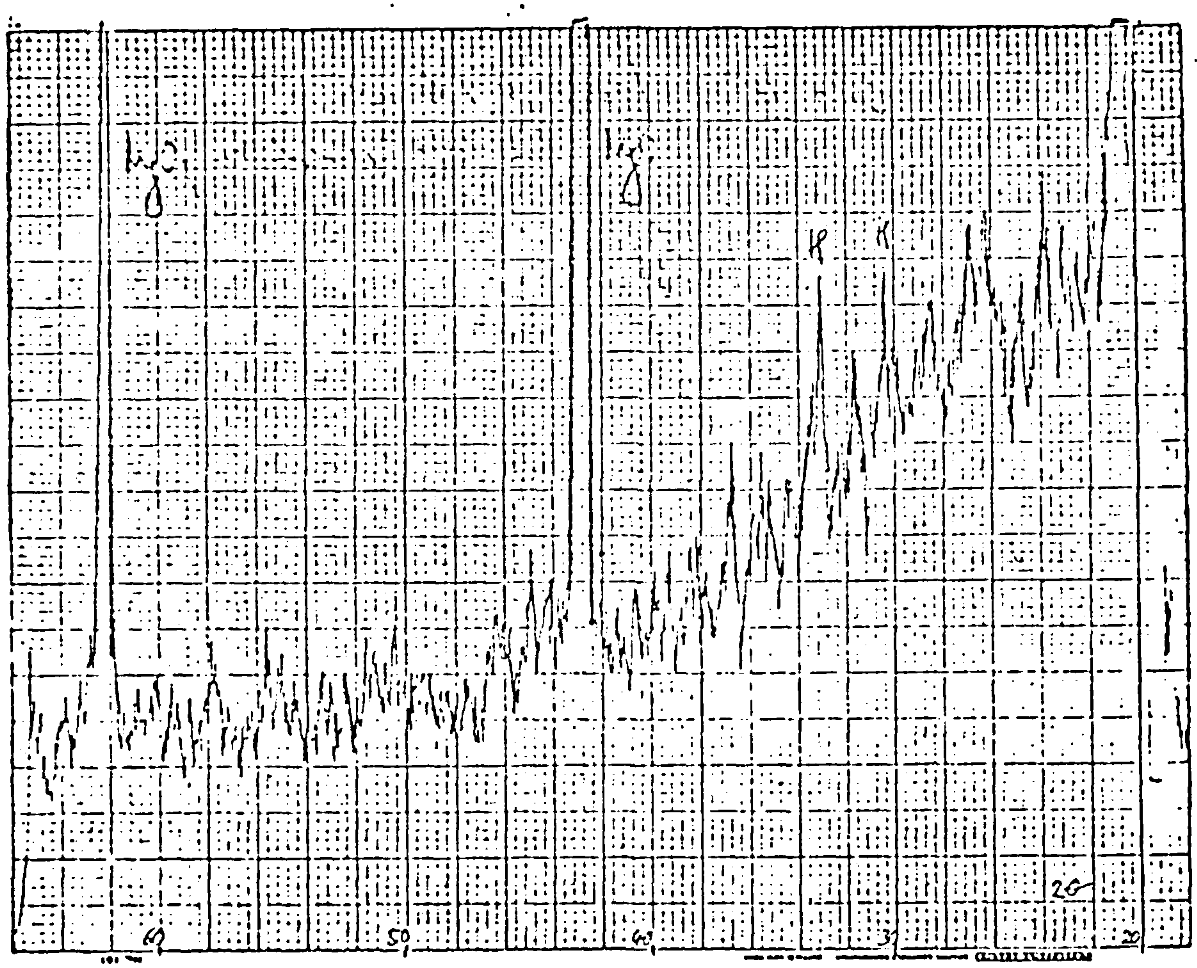

Fig. 122 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed at 100°F and cured at 30°F at 1 day.

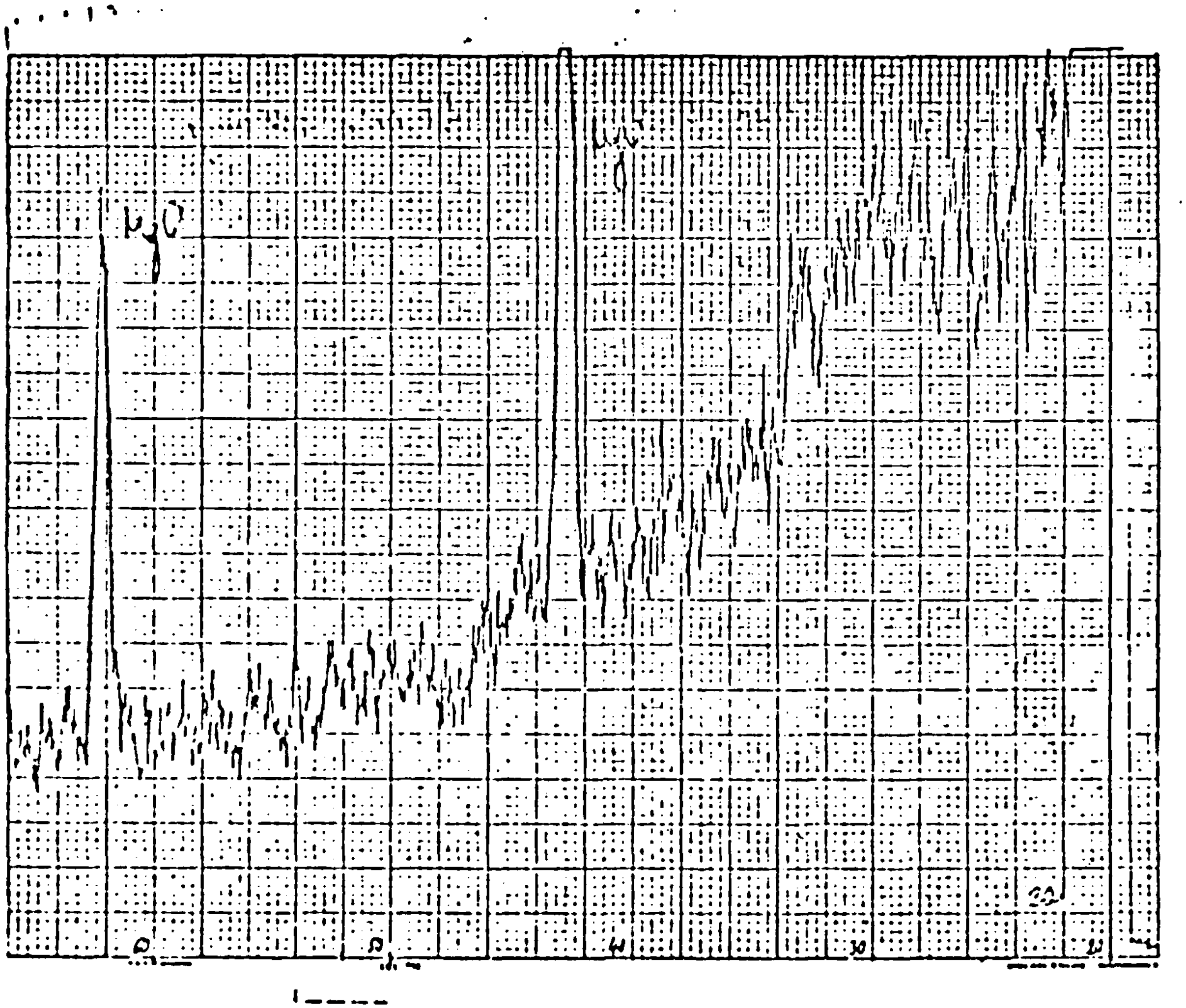

Fig. 123 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed at 100°F and cured at 30°F at 7 days.

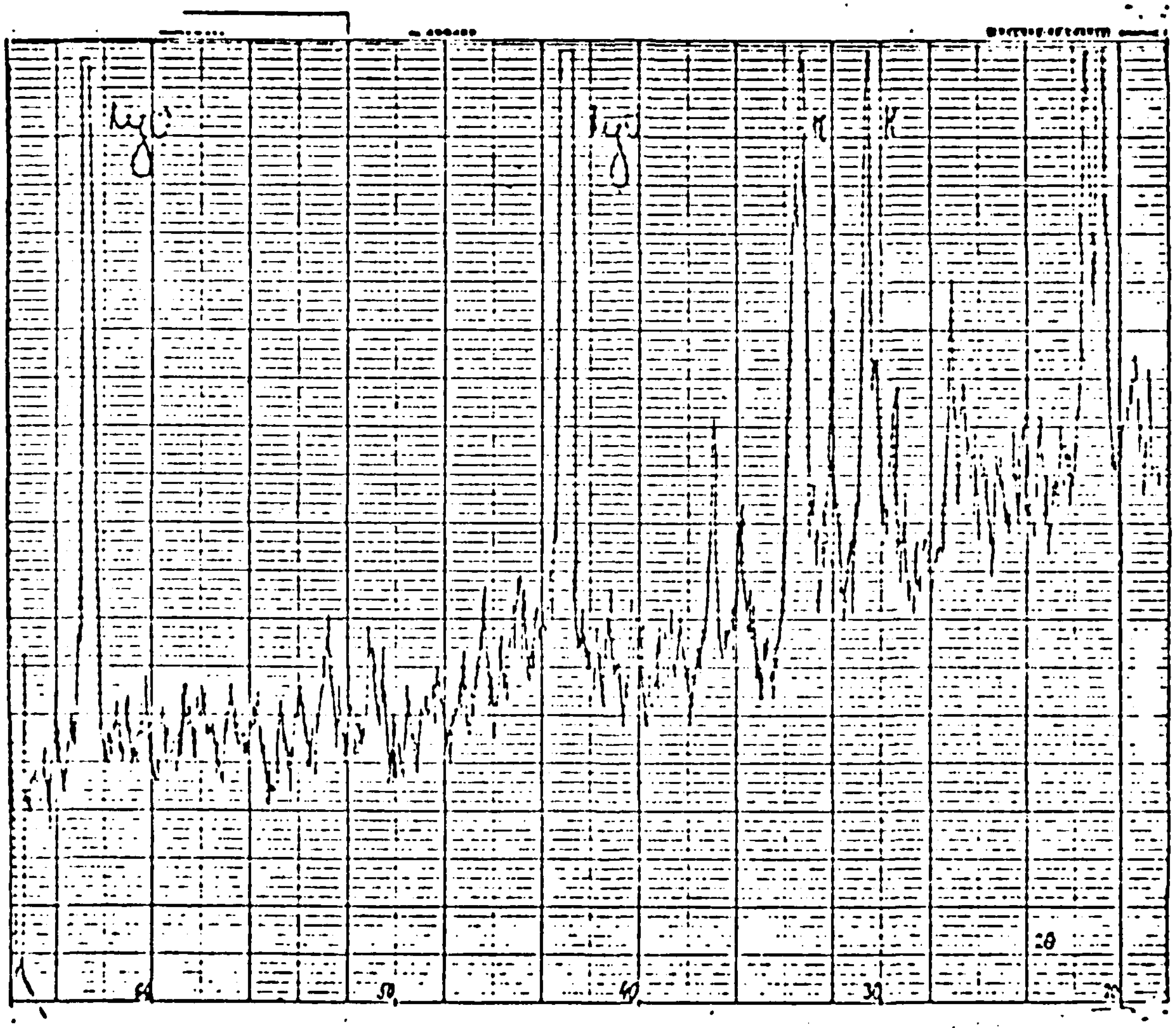

Fig. 124 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 100°F and cured at 30°F at 1 hour.

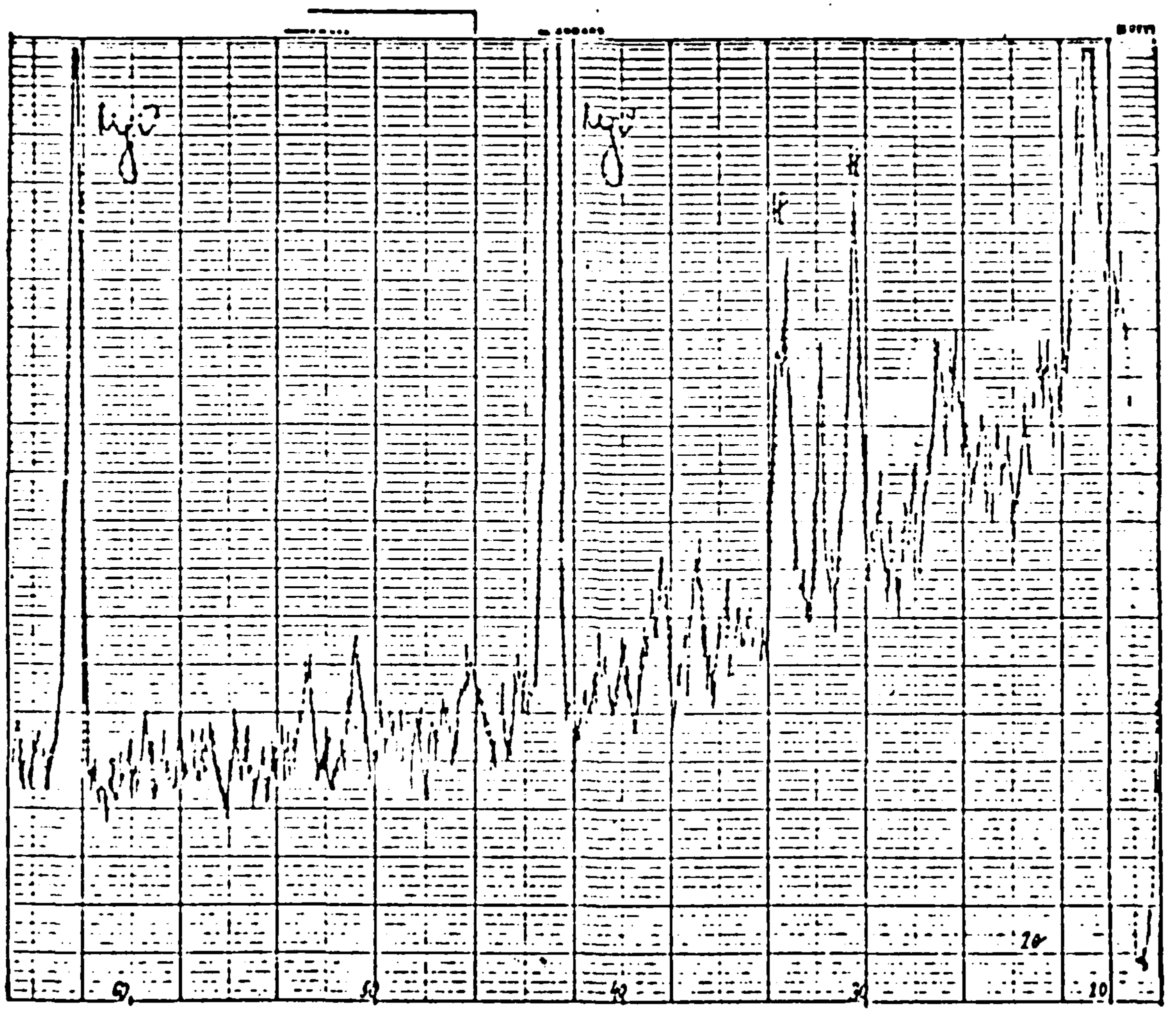

Fig. 125 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 100°F and cured at 30°F at 3 hours.

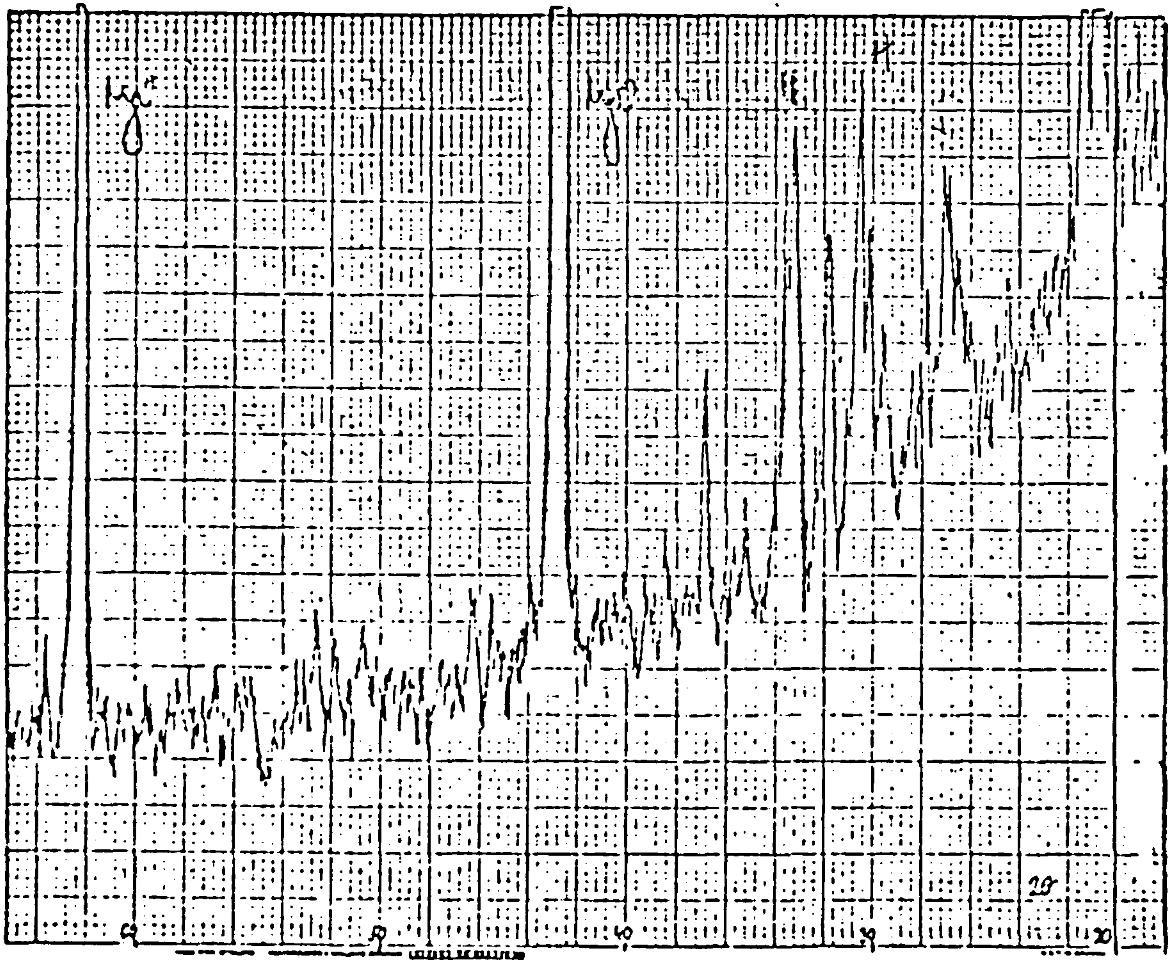

Fig. 126 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 100°F and cured at 30°F at 1 day.

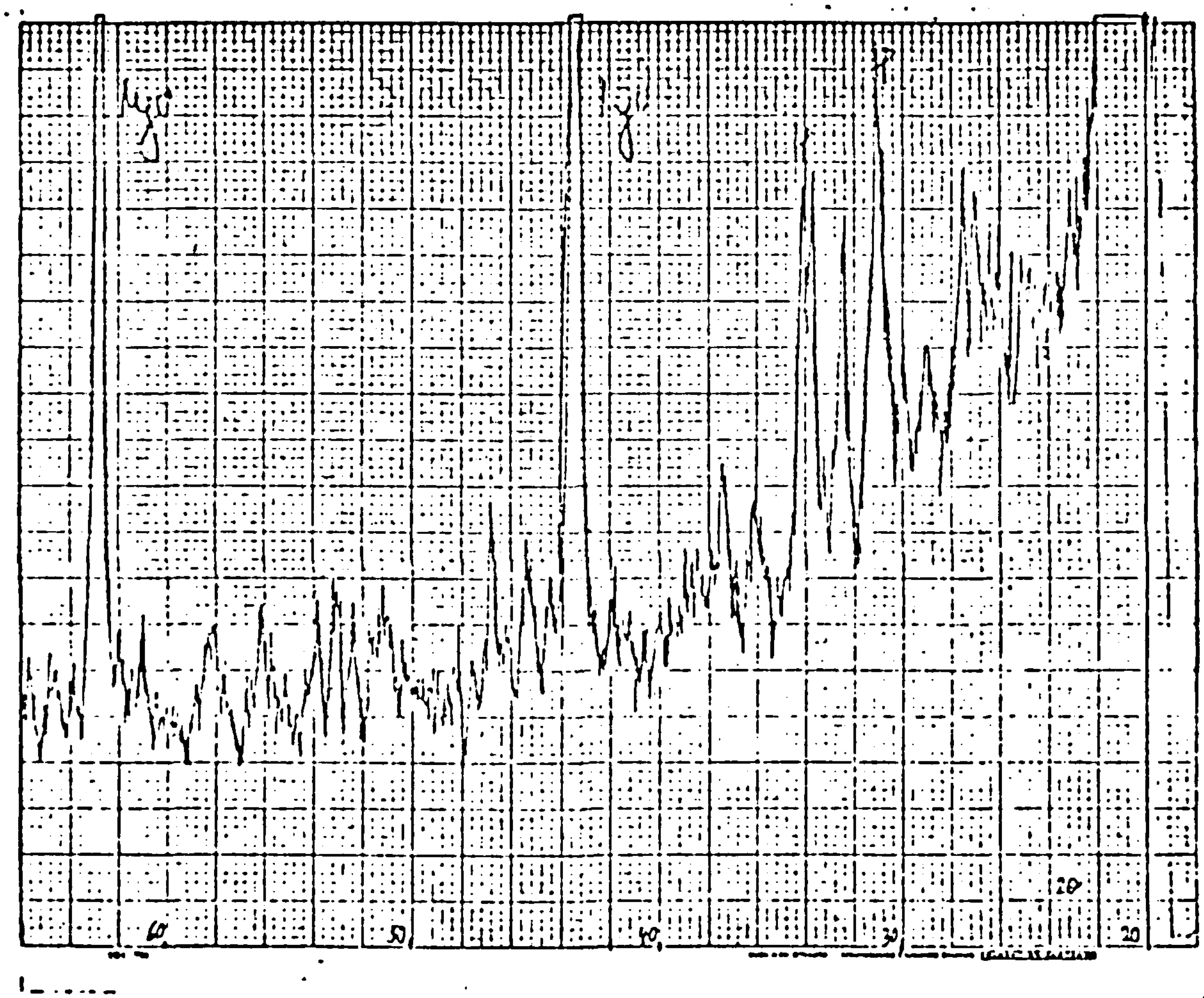

Fig. 127 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 100°F and cured at 30°F at 7 days.

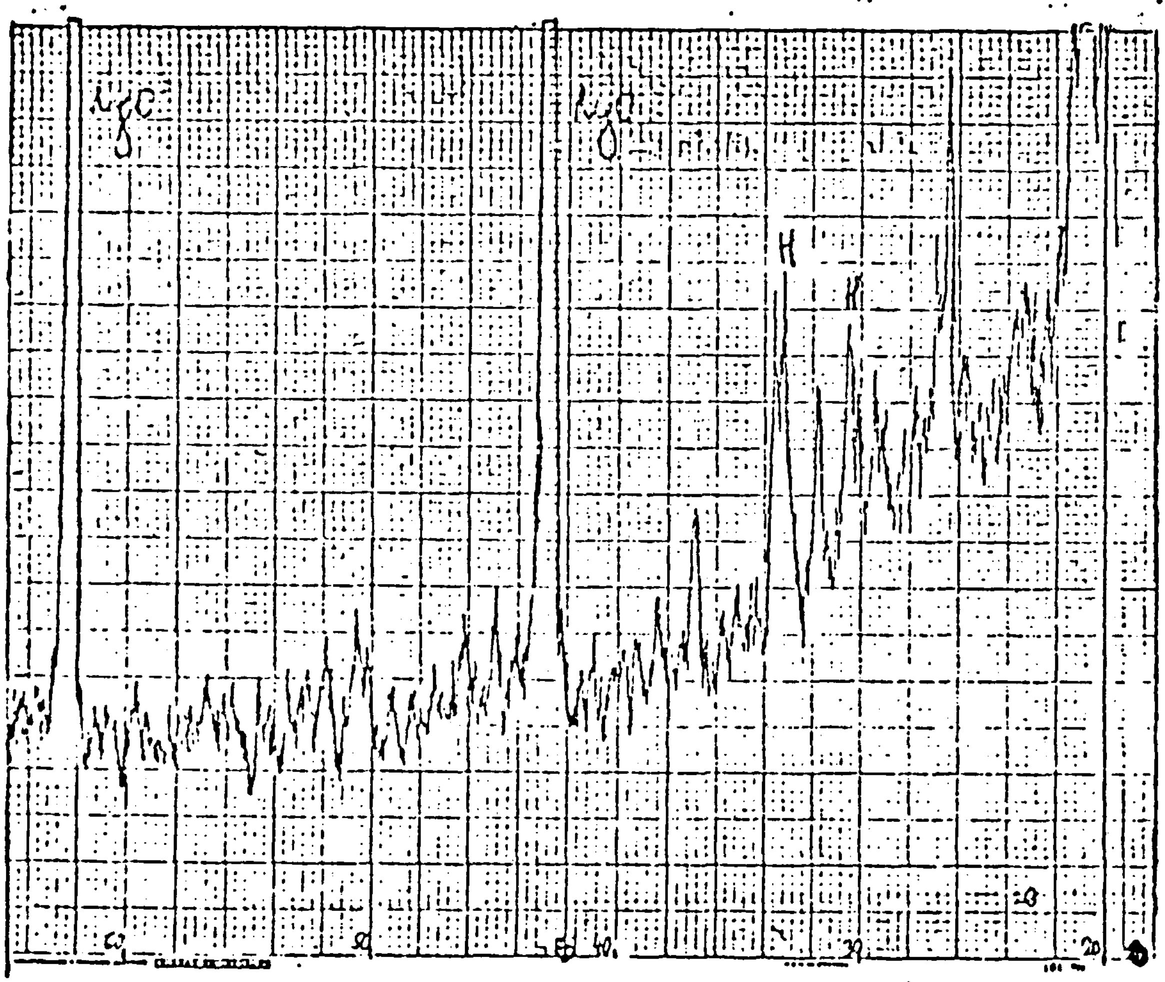

Fig. 128 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 30°F and cured at 100°F at 1 hour.

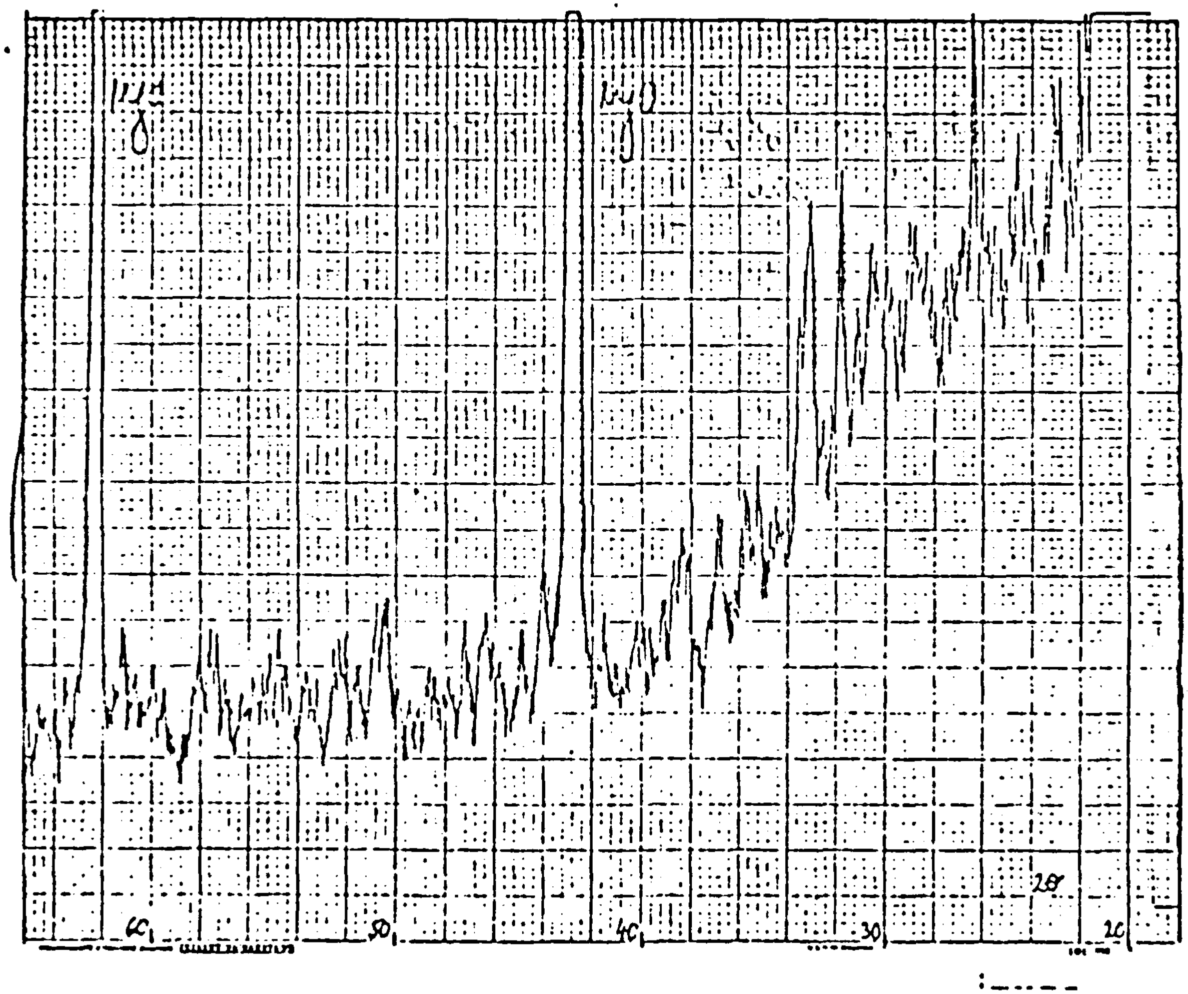

Fig. 129 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 30°F and cured at 100°F at 3 hours.

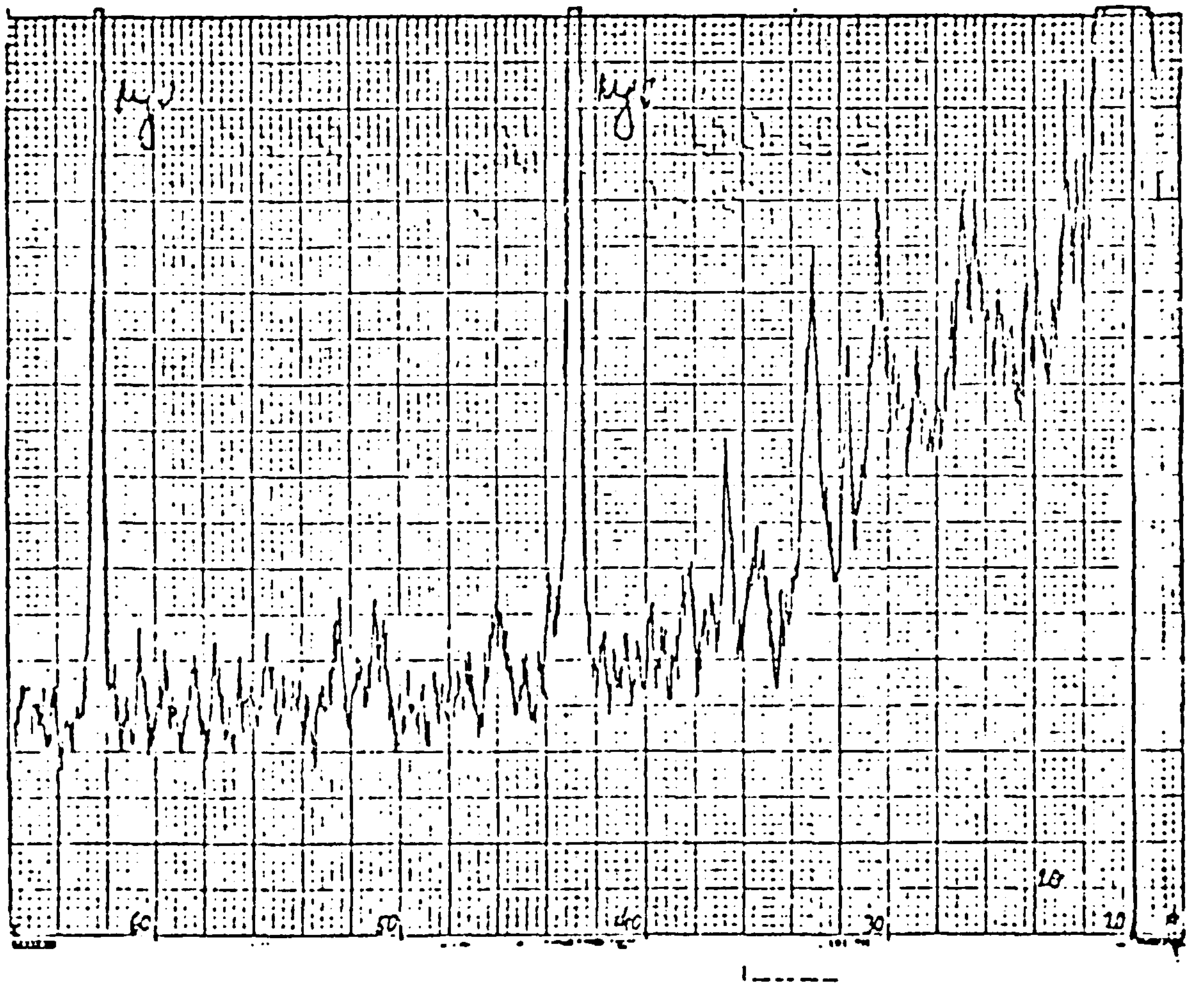

Fig. 130 - X-ray diffraction pattern of SET-45 cold weather paste mixed at 30°F and cured at 100°F at 1 day.

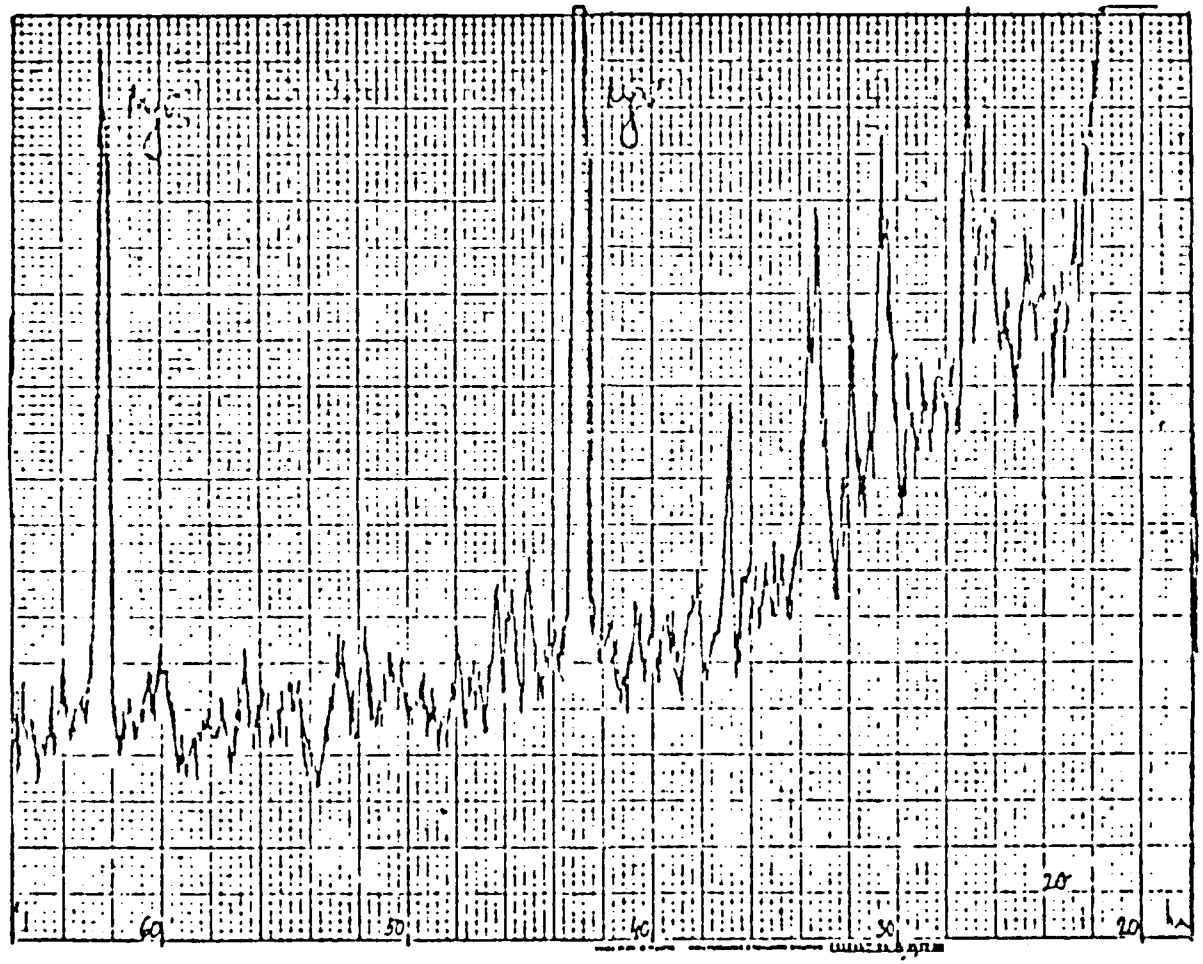

Fig. 131 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed at 30°F and cured at 100°F at 1 hour.

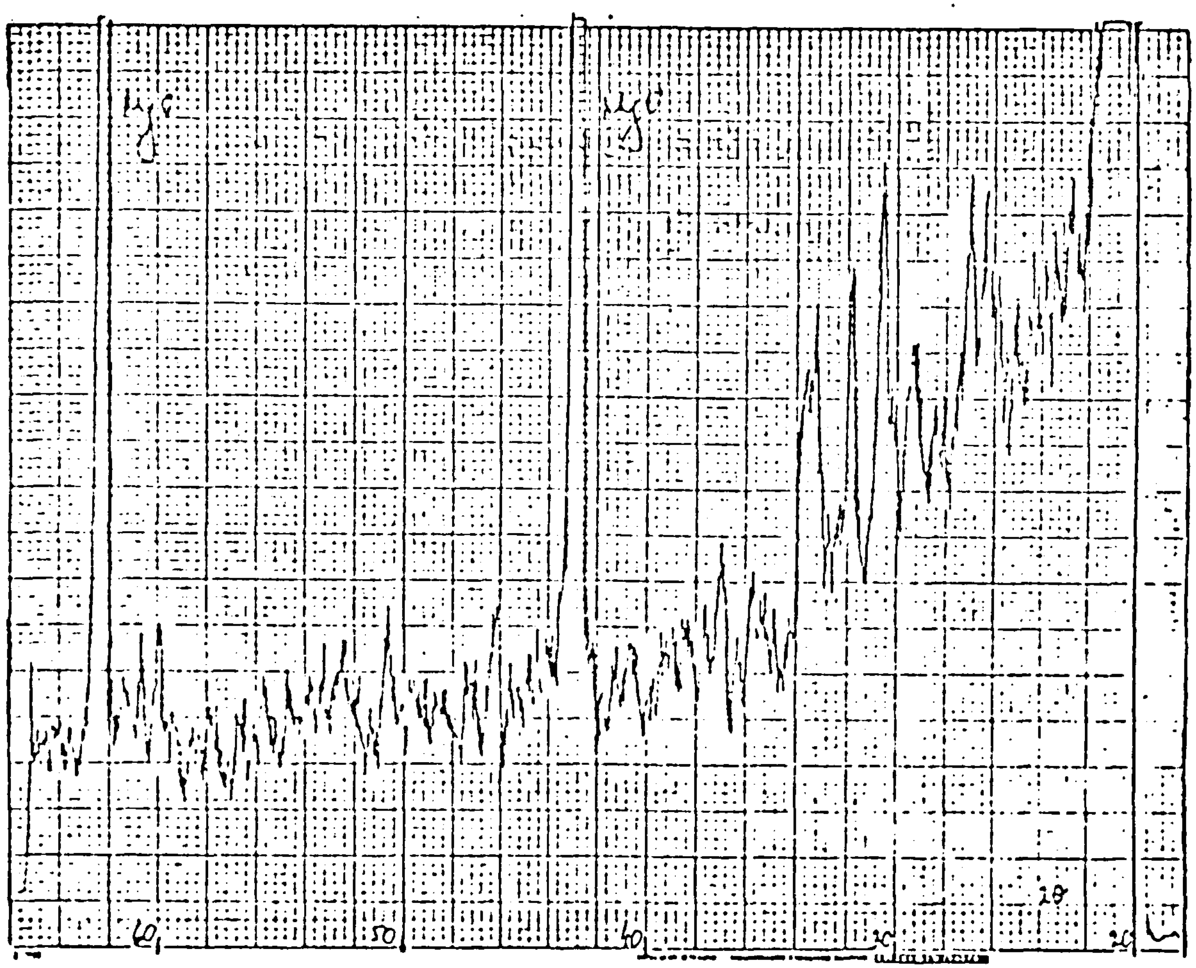

Fig. 132 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed at 30°F and cured at 100°F at 3 hours.

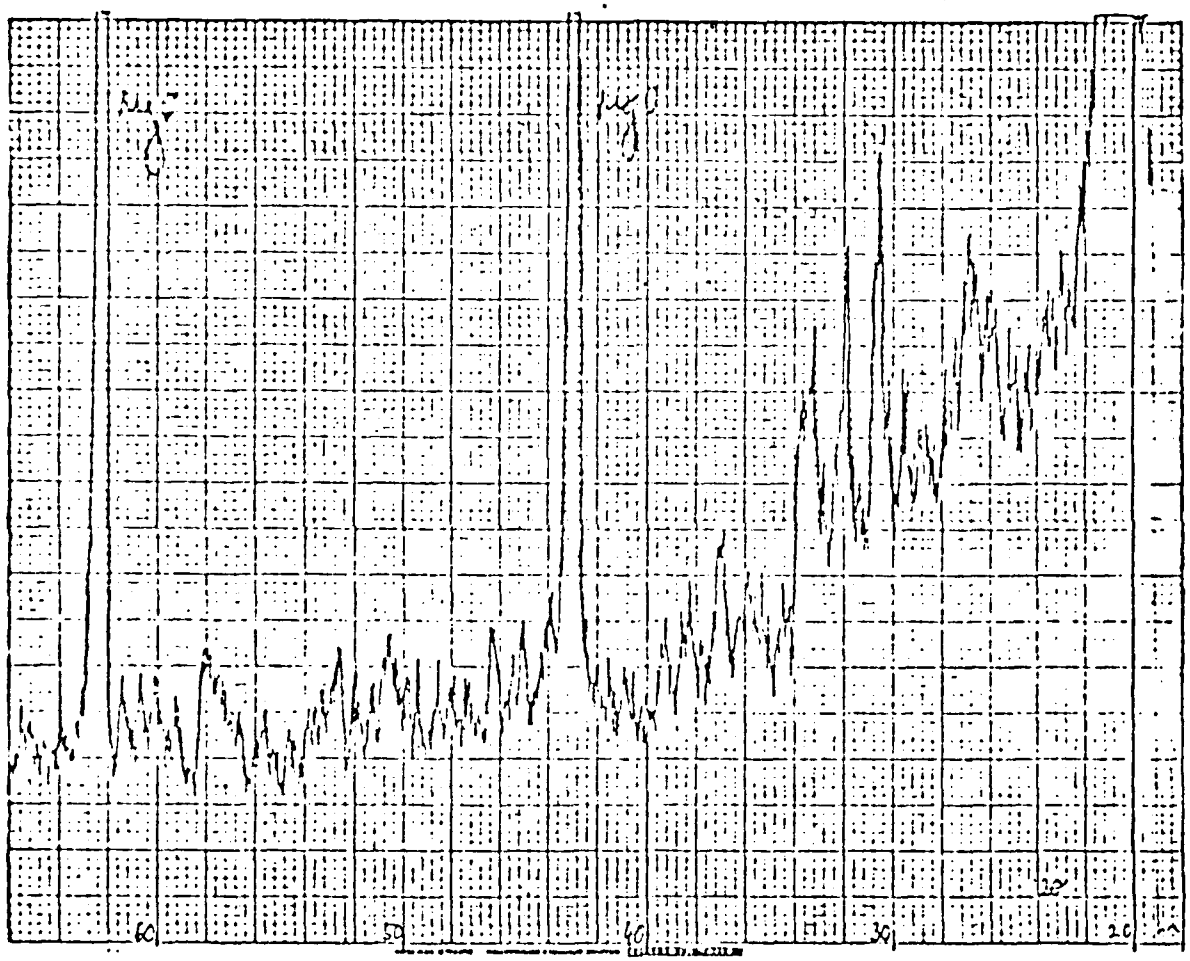

Fig. 133 - X-ray diffraction pattern of SET-45 cold weather paste with 3.5% borax mixed at 30°F and cured at 100°F at 1 day.

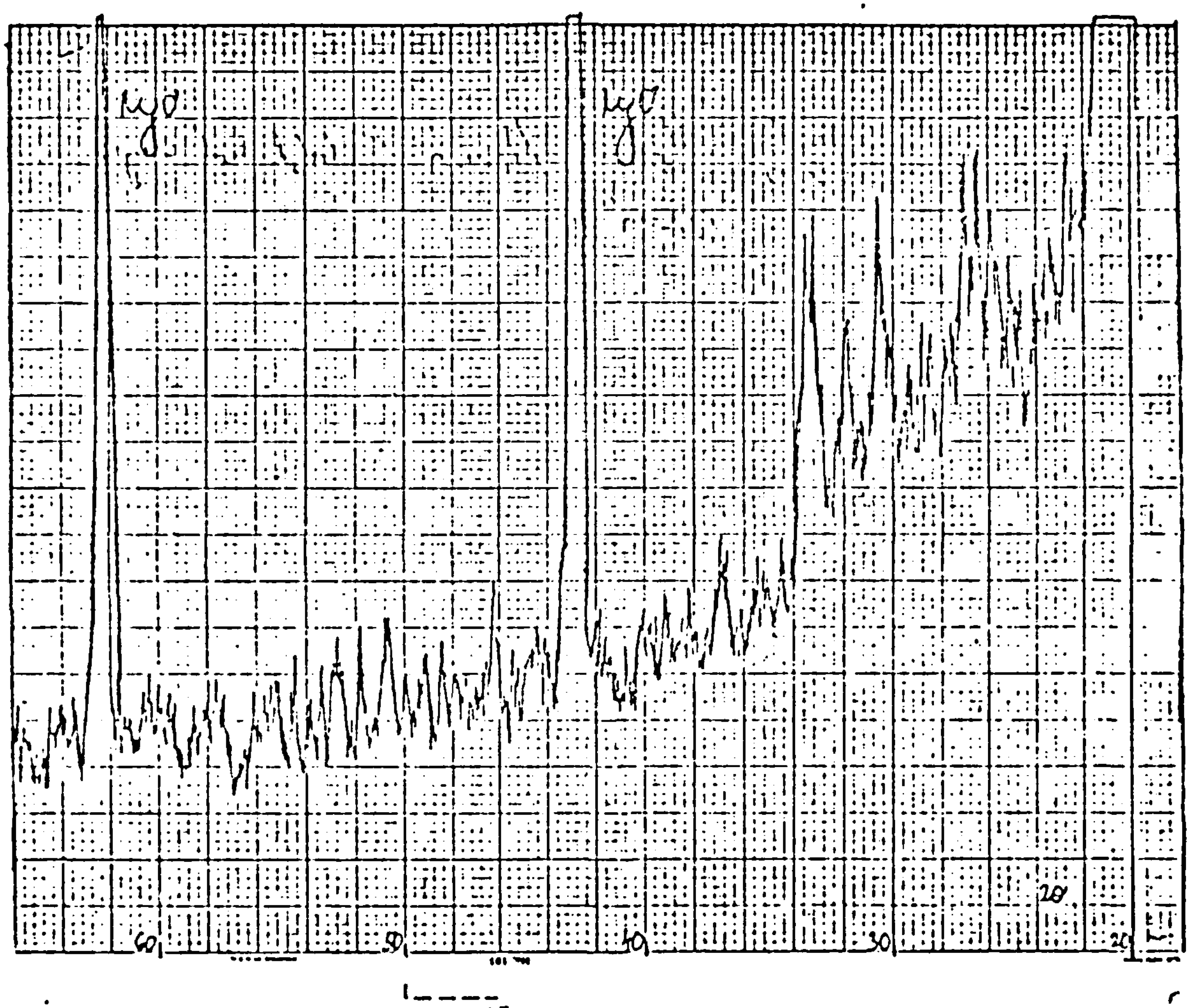

Fig. 134 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 30°F and cured at 100°F at 1 hour.

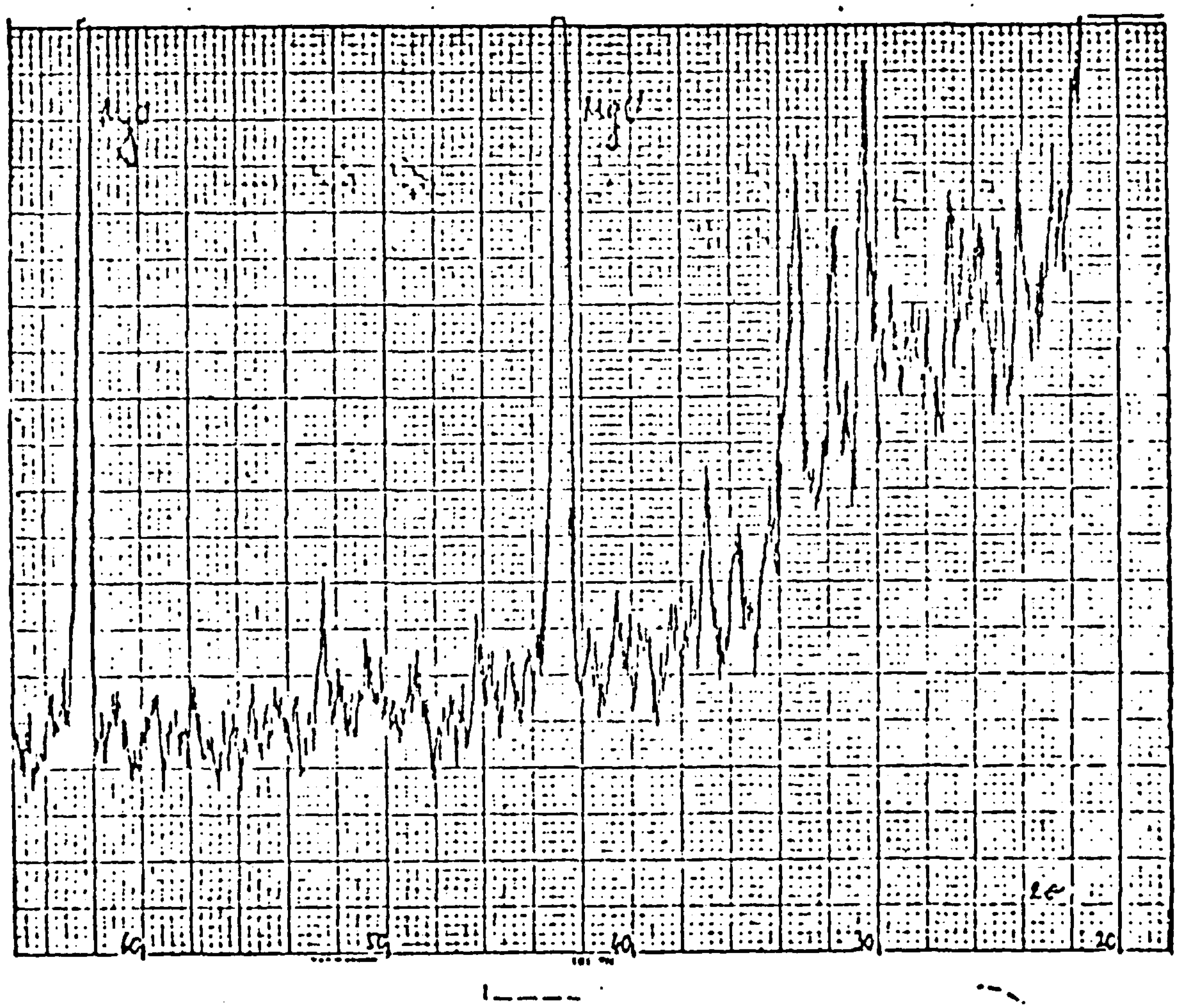

Fig. 135 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 30°F and cured at 100°F at 3 hours.

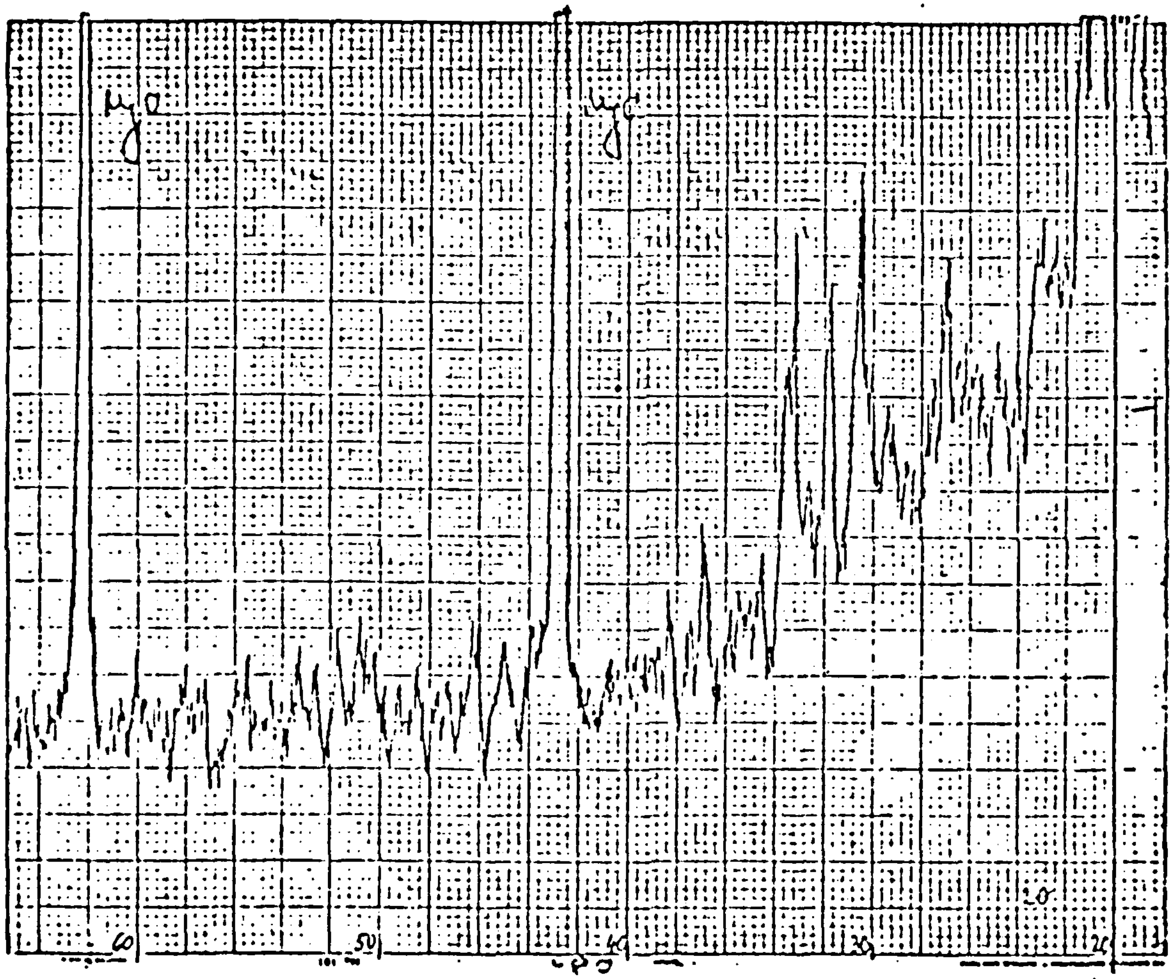

Fig. 136 - X-ray diffraction pattern of SET-45 hot weather paste mixed at 30°F and cured at 100°F at 1 day.

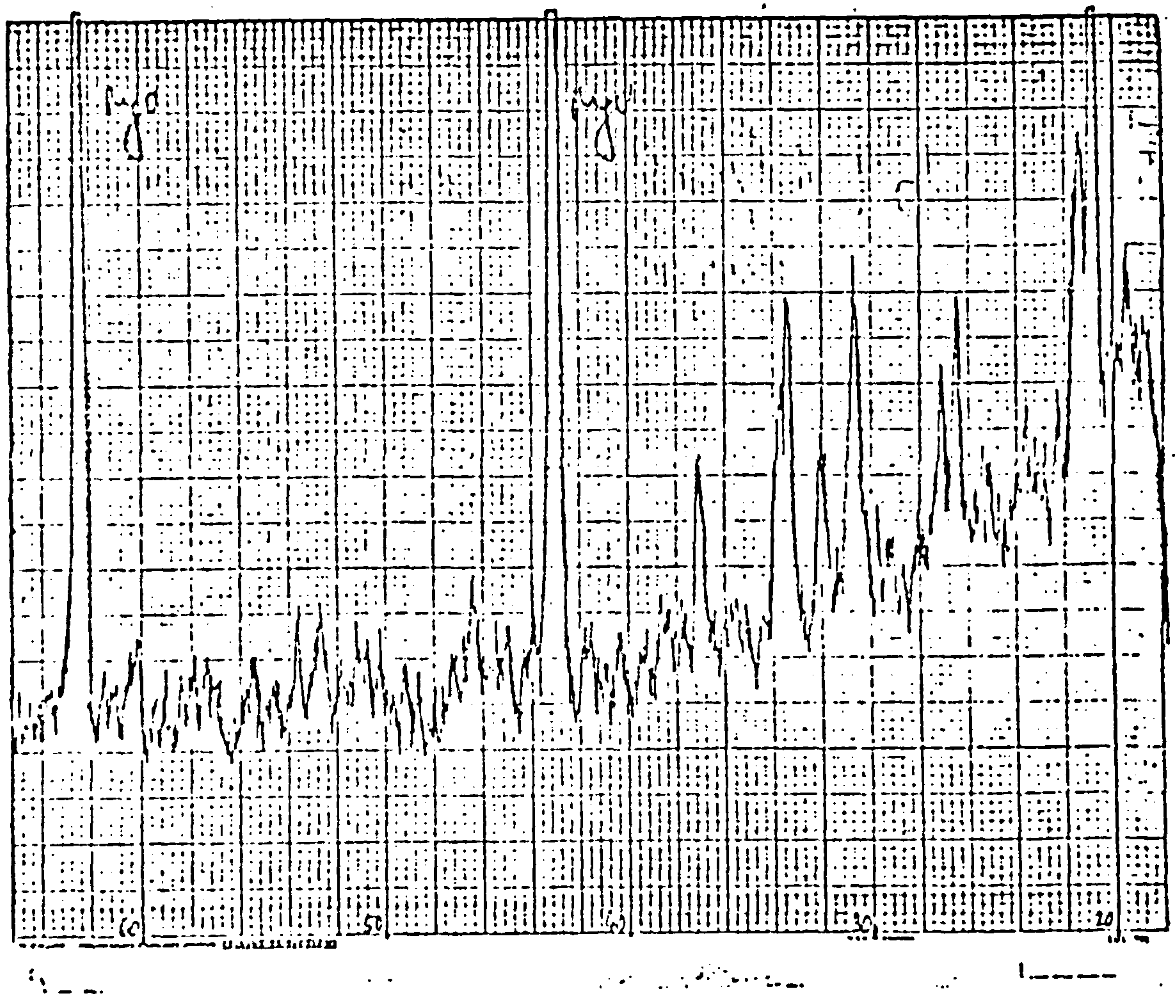

Fig. 138 - X-ray diffraction pattern of SET-45 cold:hot = 1:1 paste mixed at 30°F and cured at 100°F for 1 day.

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